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GEOCHEMICAL APPRAISAL OF EKENKPON SHALE IN THE CALABAR FLANK, SOUTHEASTERN NIGERIA

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Abstract

The hydrocarbon potential of the Cenomanian-Turonian shale units of the Ekenkpon formation in Calabar Flank was assessed via organic geochemical study using Rock Eval pyrolysis. The total organic carbon (TOC) values range from 0.33 wt. % to 2.13 wt. % (average = 0.82 wt. %) with the minimum value observed only in one of the samples (0.33). The T_{max} values obtained from the pyrolysis range from 428°C to 447°C and the calculated vitrinite reflectance (%R_o) values range from 0.54 to 0.89 indicating thermally marginal matured. Rock-Eval data revealed the organic matter to be predominantly gas prone (Type III kerogen). The values of the S_2 peak indicate poor to fair potential for oil generation. The geochemical indices suggest immature to marginally mature despite the richness of the shale units which characterized Ekenkpon type section; they have low level of conversion to generate oil at peak of maturity. The mean value of the burial temperature (T_{burial}) is 106°C suggesting that the shales are within the oil generative window.

Keywords: Ekenkpon; Hydrocarbon; Rock-Eval pyrolysis; Vitrinite reflectance; Kerogen, Burial.

1. Introduction

The Cretaceous–Tertiary succession in the Calabar Flank of southeastern Nigeria represents a post rift basin fill containing about 4km of fluvial-continental, marine and paralic sediments. The presence of associations of shales, sandstones and limestones in the area suggests possible source rocks, reservoir rocks, traps and seals which present a potential for hydrocarbon accumulation in the area. This has generated interest in the study of the regional petroleum potential of the area. The term Calabar Flank was proposed by Murat [1]. He opined that the Flank is that part of the southern Nigerian sedimentary basin characterized by crustal block faults trending in a NW-SE direction and the Calabar Flank as the Anambra sub-basin of the Benue Trough [2].

The Calabar Flank is part of the southern Nigerian sedimentary basin that is bounded by the Oban Massif to the north and the Calabar hinge line delineating the Niger Delta basin in the south and bounded the east by the Cameroun volcanic ridge [3]. The flank is one of the continental margin-sag basins which have similar features with that of Cameroon, Gabon, Congo and Angola which lies along the south Atlantic coastal margin of Africa and the origin is linked with the opening of the South Atlantic [4]. The geology of the flank shows that Calabar Flank consists of basal fluvio-deltaic grits and sandstone of the Awi Formation (Aptian-Albian); limestone and calcareous sandstones of the Mfamosing Formation (Mid-late Albian); alternating limestones and shales of the Odukpani Formation (Cenomanian), shales and marls of the Awgu Formation (Coniacian) and Carbonaceous shales of the Nkporo Shales (Campano-Maastrichtian) [5-6]. The major tectonic elements of the Calabar Flank are the Ikang Trough which for most of the depositional history was a mobile depression and the Ituk high that was a stable to somewhat mobile submarine ridge. The tectonics of the Calabar Flank is dominated by vertical movement of faulted blocks (Figure 1).

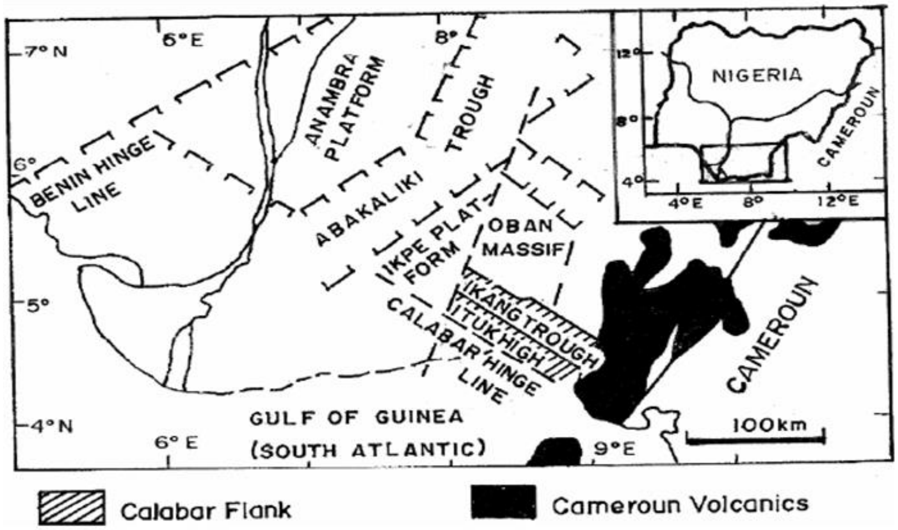

Figure 1. Map of Southern Nigeria showing Calabar Flank

The study area, Etankpini is located in Cross River within the Calabar Flank, south-eastern Nigeria between latitudes 5⁰0ʹ0ʺN and 5⁰15ʹ0ʺN and longitudes 8⁰15ʹ0ʺE and longitude 8⁰35¹0ᴵᴵ E. The study area is easily accessible by both major and minor road networks (Figure 2).

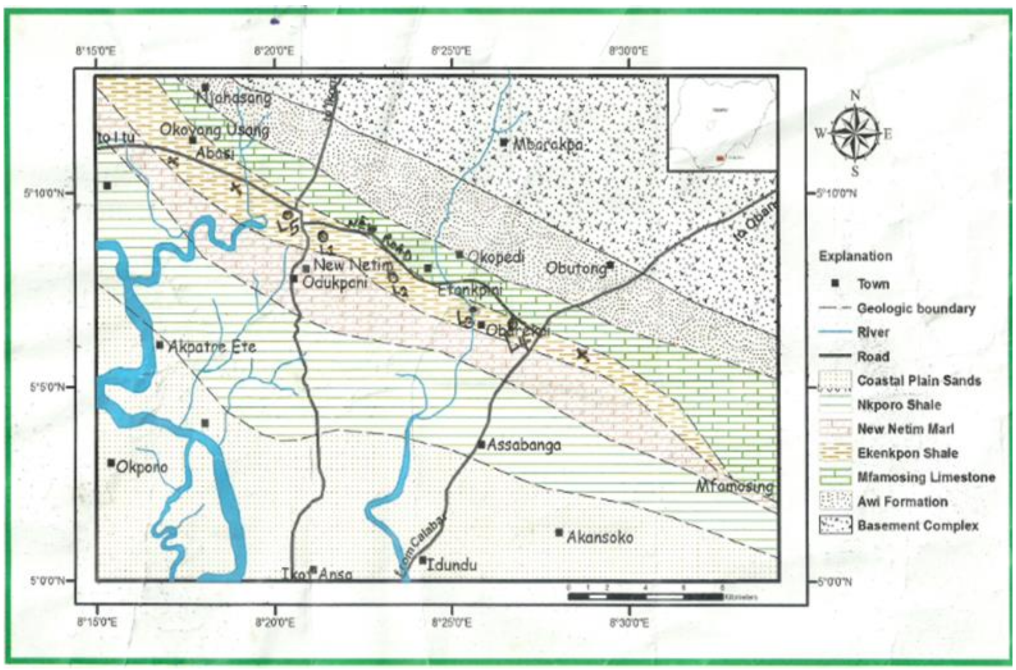

Figure 2. Geologic map showing the location, drainage and accessibility of the study area

Further previous studies on subsurface (Core) samples to better understand the petroleum play in the Calabar Flank was proposed [7] which was the main focus of this study as it attempt to characterize the Ekenkpon Shale base on the organic richness and kerogen types, variations in organic matter sources, determination of the hydrocarbon generation potential, thermal maturity and infer burial temperature.

2. Geological setting

Calabar Flank is unique being part of the southern Nigerian sedimentary basin that is bounded by the Oban massif to the north and the Calabar hinge line delineating the Niger Delta basin in the south [8]. It is also separated from the Ikpe platform to the west by a NE-SW trending fault. In the east, it extends up to the Cameroun volcanic ridge. It served as the gateway to all marine transgression into the Benue Trough and is located between two hydrocarbon provinces, the Tertiary Niger Delta and the Cretaceous Douala basin in Cameroun [9]. Structurally, it consists of basement horsts and grabens that are aligned in a NW–SE direction like other South Atlantic marginal basins in West Africa [9].

Sedimentation in Calabar Flank started with the deposition of fluvio-deltaic clastics of probably Aptian age on the Precambrian crystalline basement complex, the Oban Massif; these sediments belong to the Awi Formation [6] (Table 1 and Figure 3).

Tab. 1. Stratigraphic sequence in the Calabar flank

	AGE	FORMATION	LITHOSTRATIGRAPHIC DESCRIPTION	DEPOSITIONAL ENVIRONMENT
TERTIARY	Oligocene to recent	Benin Formation	Pebbly sands and gravels	Continental
	Eocene	Ameke Formation	Medium grained pebbly sandstones, clayey sandstones, calcareous silts, clay and thin limestone	Paralic
	Paleocene	Imo Shale	Clayey shale, clay ironstone bands, thin sandstone and sandy limestone bands	Paralic
CRETACEOUS	Maastrichtian	Nkporo Shale	Gypsiferous dark grey shales with ironstone intercalation	Shallow marine
	Campanian		Unconformity	
	Santonian Coniacian	New Netim Marl	Marlstone with shale intercalations	Marine
	Turonian Cenomanian	Ekenkpon Shales/Nkalagu Formation	Thick black pyritic shales with intercalations of mudstones, sandstones, ironstones and oyster beds	Marine
	Albian	Mfamosing Limestone	Stromatolitic fossiliferous limestones	Marine
	Aptian	Awi Formation	Arkosic sandstones interbedded with shales	Fluvio-deltaic
	Precambrian	Oban Basement Complex	Crystalline basement rocks	

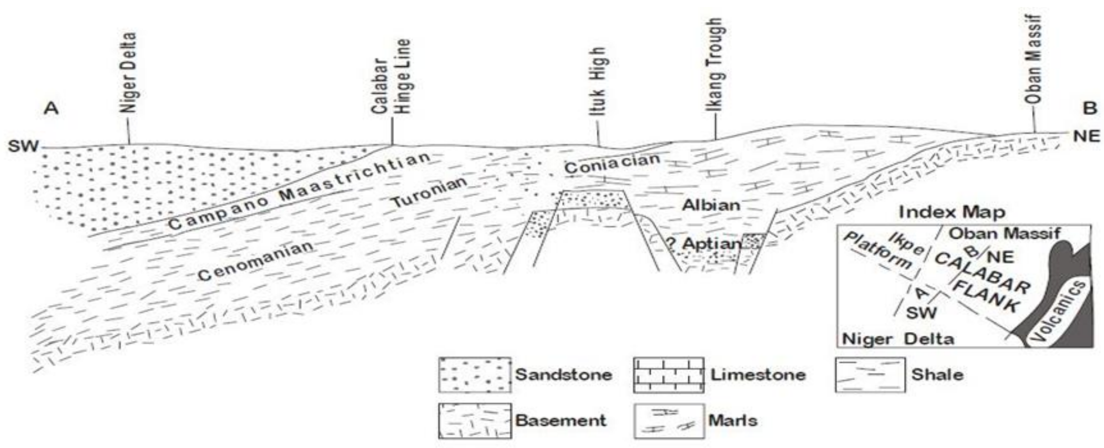

Figure 3. Structural elements and conceptual subsurface distribution of Cretaceous sediments in the Calabar Flank [15]

The earliest marine transgression into the Calabar Flank occurred in the Mid-Albian with the deposition of platform carbonate of the Mfamosing Limestone deposited in diverse depositional environments. The Mfamosing Limestone is overlain by a thick sequence of black to gray shale unit, the Ekenkpon Formation [10]. This formation is characterized by minor intercalation of marls, calcareous mudstone and oyster beds. This shale unit was deposited during the late

Cenomanian-Turonian. The Ekenkpon Shales are overlain by a thick marl unit, the New Netim Marl, characterized with nodular structures, shaly at the base and interbedded with thin layer of shales at the upper-section [10]. Foraminiferal and coccolith evidence suggest early Coniacian age for this marl unit [8, 11]. The New Netim marl is unconformably overlain by carbonaceous dark grey shale of Nkporo Formation [12]. This shale unit was deposited during the late Campano–Maastrichtian. The Nkporo Shale caps the Cretaceous sequence in the Calabar Flank which is overlain by a pebbly sandstone unit of the Tertiary Benin Formation. The Calabar Flank is a sedimentary basin that borders southeastern Nigeria's continental margin. The Calabar Flank is at right angles to the major rift faults of the Benue Trough and structurally consists of NW-SE trending basement horsts (the Oban massif and the Ituk-high) separated by a graben and the Ikang trough [13]

3. Materials and methods

A total of forty (40) subsurface (Core) samples were retrieved from two development wells with a total depth of 60m within the area. The outcrops visited were five different road cut exposures of shale showing different features such as septarian nodules, and fissility (Figure 7). Most of the shale samples are grey to dark grey in colour. The main lithology is the dark grey fissile shale intercalated with light grey band of mudstone with the thickness of the exposures ranging from 5.6m to 11.7m. The two wells studied (Well 13 and Well 14) penetrated the Ekenkpon Formation within the Calabar Flank. The samples were retrieved from 24m, 38m, 50m, 56m, 60m (well 13) and 29m, 35m, 50m and 56m (well 14) respectively. Twenty samples obtained from each well were sampled at 3m interval. Ten samples were systematically selected from the two wells for organic geochemical analyses. The samples were dried, pulverized and sent for analysis at Weatherford Laboratories, Texas, USA (Figures 5-7). They were treated with concentrated HCl to remove carbonates and total organic carbon (TOC) is measured using LECO 230 analyzer. All samples were subjected to Rock-Eval pyrolysis, according to the standard procedures with the use of Rock-Eval II instrument. Parameters such as the TOC, S_1 (hydrocarbons released at temperatures of about 300°C), S_2 (hydro-carbons generated by pyrolytic degradation of organic matter at temperatures ranging from 300°C-550°C), S_3 (CO_2 generated during thermal cracking of the kerogen); T_{max} (temperature at which the maximum amount of hydrocarbons are generated); hydrogen index ($HI = S_2 \times 100/TOC$), oxygen index ($OI = S_3 \times 100/TOC$), production index ($PI = S_1/(S_1 + S_2)$), normalized oil content ($S_1 \times 100/TOC$), and calculated vitrinite reflectance (Calc. $\%R_o = 0.0180 \times T_{max}$).

4. Results and discussions

4.1. Quantity of organic matter

The TOC indicates the richness of the organic matter in the rock and it includes both the insoluble organic matter (kerogen) and the soluble organic matter (bitumen). The TOC and Rock-Eval pyrolysis were carried out on selected screened rock samples (Tables 2 and 3) [5-11, 14].

The results of the samples correlated with the earlier study on surface samples from this least investigated area [4]. The TOC values showed that the beds are organically rich, except for D2 sample.

The samples exhibit fair to good source rocks except sample D2 (TOC = 0.33 wt. %) which does not exhibit minimum threshold value (0.5wt.%) for potential source rocks (Tables 2) [14-21]. The TOC of Ekenkpon Shale range between 0.33 wt. % and 2.13 wt. % (average=0.82wt. %) with the minimum value in sample D2 (0.33wt. %). These are indicative of poor to good potential source rocks [22-23]. The vertical and lateral variations in organic matter could be attributed to localized changes in organic productivity and preservation as well as differential depositional environments. The pyrolysis indices of Ekenkpon Shale show S_1 values to range from 0.02 - 0.45; S_2 values range from 0.29 to 3.9 while all the samples have S_3 values less than 0.5 suggesting poor to good generative potential. The low to moderate genetic potential, S_1+S_2 (GP), ranging from 0.32mg/g rock to 4.35mg/g rock indicate a gas prone to moderate source rock with poor to fair oil potential. These low values suggest that the S_1 and S_2 generally indicated initially poor generative potential due to a large proportion of woody (humic) and oxidized kerogen. The S_1 values could have been lowered by oxidation and by adsorption on clay minerals of the hydrocarbon produced during pyrolysis. The Hydrogen Index (HI) values are generally low (< 200) for all the samples (Table 2) [14–22].

Table 2. Total Organic Carbon, Programmed Pyrolysis Data of the Ekenkpon Shales

Sample No	Depth (feet)		Sample Type	Leco TOC	SRA			T_{max} (°C)	Calc. %R_o based on T_{max}	HI	OI	S_1+S_2	S_2/S_3	S_1/TOC *100	PI
	Top	Bottom			S_1	S_2	S_3								
D1	-	-	Powder rock	0.56	0.03	0.37	0.14	431	0.60	66	25	0.4	3	5	0.07
D2	-	-	Powder rock	0.33	0.03	0.29	0.14	428	0.54	88	42	0.32	2	9	0.09
D3	-	-	Powder rock	0.55	0.02	0.38	0.10	430	0.58	69	18	0.4	4	4	0.05
D4	-	-	Powder rock	2.13	0.45	3.90	0.21	437	0.71	183	10	4.35	19	21	0.10
D5	79 (23.9m)	89 (27m)	Powder rock	0.57	0.05	0.37	0.07	438	0.72	65	12	0.42	5	9	0.12
D6	148 (44.8m)	157 (47.6m)	Powder rock	0.52	0.04	0.33	0.07	434	0.65	63	13	0.37	5	8	0.11
D7	178 (53.9m)	188 (57m)	Powder rock	0.60	0.04	0.43	0.15	441	0.78	72	25	0.47	3	7	0.09
D8	89 (27m)	99 (30m)	Powder rock	1.10	0.11	1.04	0.15	447	0.89	95	14	1.15	7	10	0.10
D9	118 (35.8m)	128 (38.8m)	Powder rock	0.64	0.05	0.33	0.18	442	0.80	52	28	0.38	2	8	0.13
D10	158 (47.9m)	168 (50.9m)	Powder rock	1.20	0.12	1.06	0.16	434	0.65	88	13	1.18	7	10	0.10

Note

TOC – Total Organic Carbon, wt%
S_1 - Volatile hydrocarbon (HC) content, mg HC/g rock
S_2 – remaining HC generative potential mg HC/g rock
S_3 – carbon dioxide content, mg CO_2/g rock
TOC on LECO Instrument
SRA- Programmed pyrolysis on SRA instrument
RE – Programmed Pyrolysis on Rock-Eval instrument
NOPR – Normal Preparation
**- sample contaminated*
***- low S_2, Tmax is unreliable*
Meas. % Ro – measured vitrinie reflectance
HI – hydrogen index= S_2 x 100/TOC, mg HC/g TOC
OI – oxygen index= S_3 x 100/TOC, mg CO2/g TOC
PI- Production index= S_1/(S_1+S_2)
EXT – Extracted Rock

Table 3. Geochemical parameters showing level of thermal maturation and estimated burial temperature

Sample no	This study				Peters [17] and Baker [2]				
	%Ro	Burial temp., °C	PI	T_{max}, °C	Maturation	PI	T_{max}, °C	%Ro	Burial temp., °C
D1	0.598	92	0.075	431	Top of oil generative window	0.1	435-445	0.6	93
D2	0.544	82	0.09375	428					
D3	0.58	89	0.05	430					
D4	0.706	110	0.103448	437					
D5	0.724	112	0.119048	438	Bottom of oil generative window	0.4	470	1.4	181
D6	0.652	101	0.108108	434					
D7	0.778	120	0.085106	441					
D8	0.886	133	0.095652	447					
D9	0.8	123	0.091264	442					
D10	0.65	101	0.131421	434					
Mean	0.6918	106	0.10453	436					

The distribution of the shale samples in the plot of hydrocarbon potential and TOC shows Type III kerogen which is gas prone. However, variations of this parameter within this sample could be attributed to differences in organic facies. The low HI values are dominated by type III kerogen predominantly of terrestrial plants. Overall low HI and S_2/S_3 values (Table 2) indicate that the Ekenkpon Shale samples are mainly gas prone. The hydrocarbon potential (S_2) values for Ekenkpon Shale vary from 0.29 to 3.90 depicting poor to fair potential to generate oil [22-23].

The S_3 values range from 0.07 to 0.21, this is the carbon dioxide evolved during pyrolysis. The S_2/S_3 ratio range from 2.0 to 19.0 for D1, D2, D3, D7 and D9 samples, however, values less than 5 suggest Type III kerogen which could generate only gas at peak maturity (Table 2). Consequently, samples D5, D6, D8 and D10 values suggest Type II/III kerogen which could generate oil and gas at peak maturity. For sample D4, S_2/S_3 value is greater than 15 implying Type I kerogens which is oil prone at peak maturity.

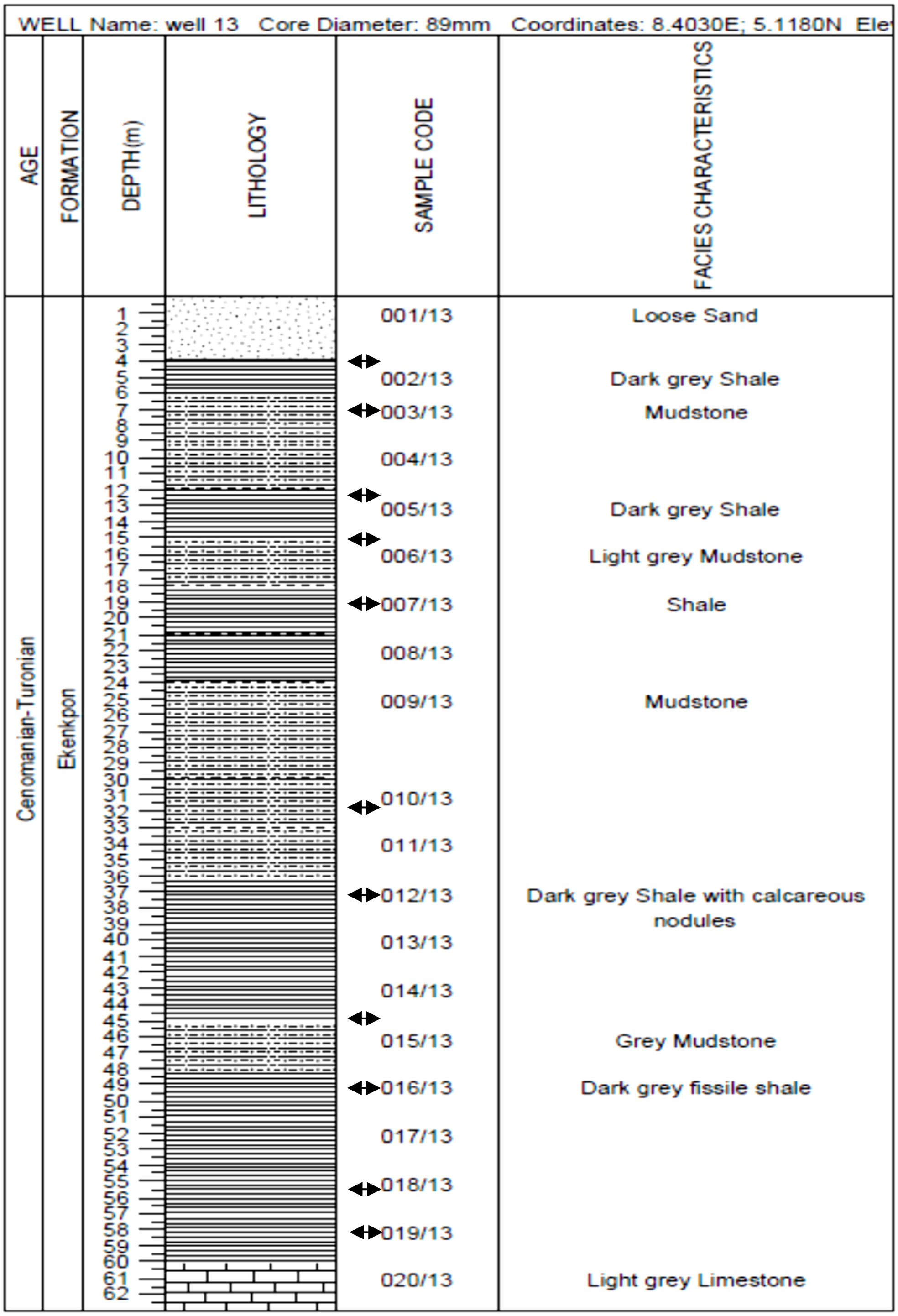

Figure 4. Lithologic Log description of well 13

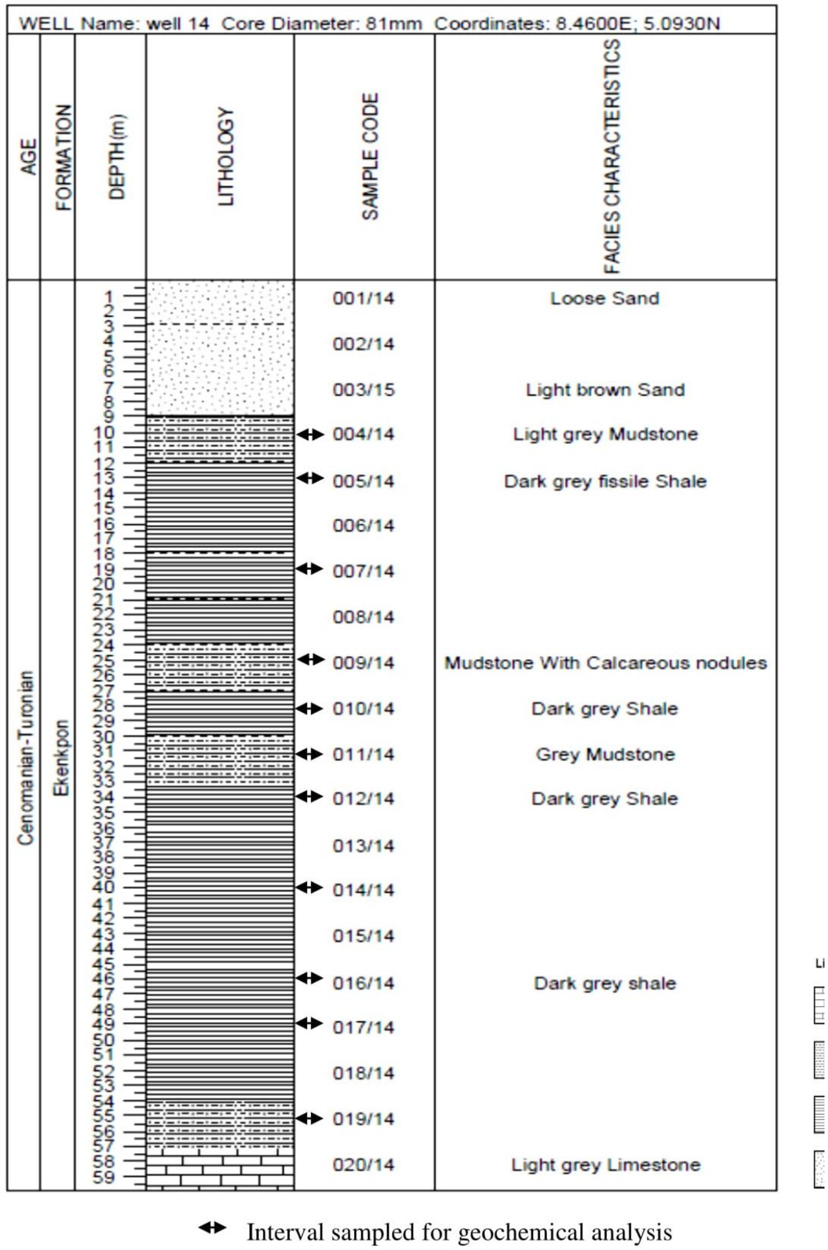

Figure 5. Lithologic Log description of well 14

4.2. Types of organic matter

The organic matter types in the sediments were assessed by Rock-Eval pyrolysis (Table 2). Most of the studied rock units from the core samples are mainly Type III with subordinate Type II/ III (Figures 7-11) [14-20,24]. The T_{max} values from the previous study range from 428°C to 437°C, while this study showed T_{max} values to range from 428°C to 447°C. The correlated values from surface samples [7] and this study (Core samples), indicate immature to marginally mature source rocks which were also evident in the core samples.

The HI values of Ekenkpon shale are below 300 mg HC/g TOC indicating Type II/III kerogen, this has a relatively high H/C ratio (1.0 to 1.4) and a low O/C ratio (0.09 to 1.5) (Table 2). Only sample D4 has HI value close to 200 mg HC/g TOC (HI=183 mg HC/g TOC) while others are very low. It is a good oil or gas prone kerogen. Most of the HI in Ekenkpon Shale falls within the range of Type III kerogen which is less favourable for oil generation, but gas.

A wide range in kerogen compositions also is suggested by variations in HI and S_2/S_3 ratio. HI values range from 52 to 183. Seven of the Ekenkpon shale samples exhibit HI values less than 150 and values of S_2/S_3 less than 10 suggesting gas generative potential (Tables 2) [14-20].

Majority of the samples plotted along the Type II and Type III evolution plot of HI versus OI (Figure 9) suggesting oil and gas prone source rock. A plot of the Hydrogen Index HI versus T_{max} also shows that the samples contained mixed Type II and Type III organic matter (kerogen) (Figures 8-11).

Figure 6. Road cut exposure of Ekenkpon Shale on the New Etankpini road showing a close view of the septarian nodule

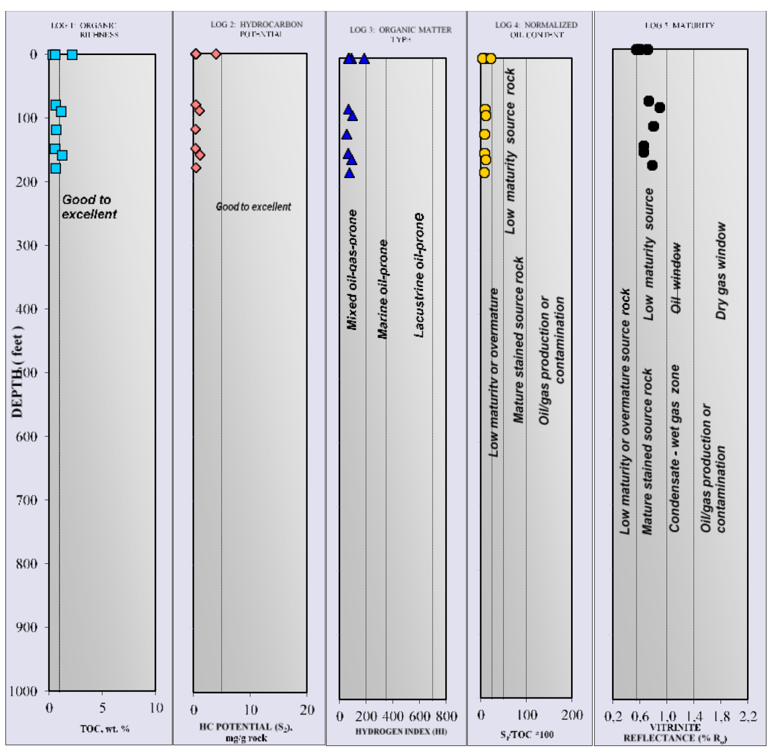

Figure 7. Geochemical logs of the studied wells

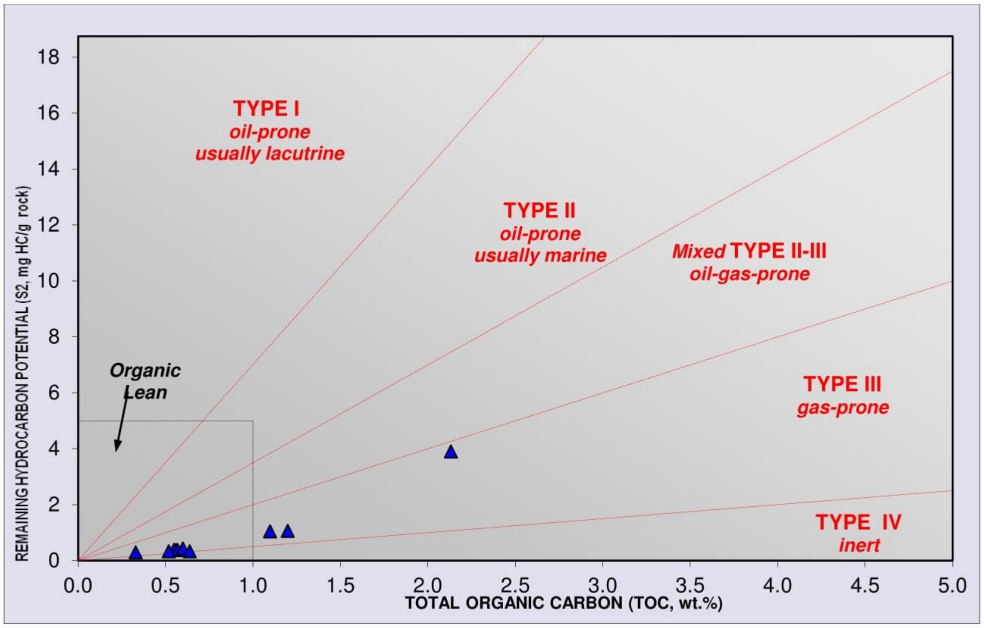

Figure 8. Hydrocarbon potential (S2) vs TOC showing the dominance of Type III kerogen in Ekenkpon Shales

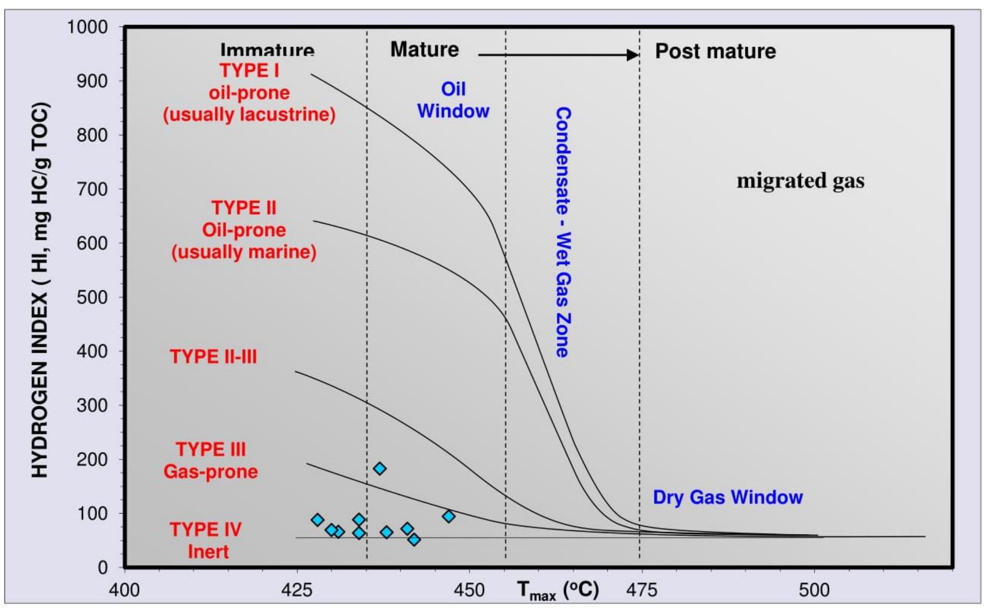

Figure 9. Plot of HI versus T_{max}(°C)

4.3. Thermal maturation of the organic matter

Thermal maturity provides an indication of the maximum palaeo-temperature reached by a source rock. The thermal maturity of the Ekpenkon shale of the Calabar Flank has not been widely discussed. Since the thermal maturity (T_{max}) of organic matter is indicated by vitrinite reflectance values, the calculated vitrinite reflectance (%R_0) values for the Ekenkpon shale range from 0.54 to 0.89 which suggest immature to early or marginally mature source rock [25] with the mean vitrinite reflectance value of 0.68% (Table 3).

The production index (PI) values range from 0.05 to 0.13. A plot of PI vs T_{max} indicates low level conversion and the T_{max} values range from 428 to 447°C for all the samples (Figure 12, Table 2). Four of the sample analyzed showed T_{max} values lower than 435°C suggesting thermally immaturity with respect to the oil generative window, while the remaining samples are thermally mature. The low T_{max} values can be caused by could be due to occurrence of resinite (fossil tree resin) and by the oil that has been generated in place or migrated into the rock [26]. The PI versus T_{max} plots indicates low level of conversion for some of the samples while others are immature and some are within the oil window (Figures 12 and 13). Calculated R_o values (0.54-0.89) show that the Ekenkpon Shales have low thermal maturation, indicating that, in this area, this formation has not yet entered the oil window [14-21,24] (Table 2 and Figure 11), as a consequence, any gas present in this area (Ekenkpon Formation) must have been generated in association with oil.

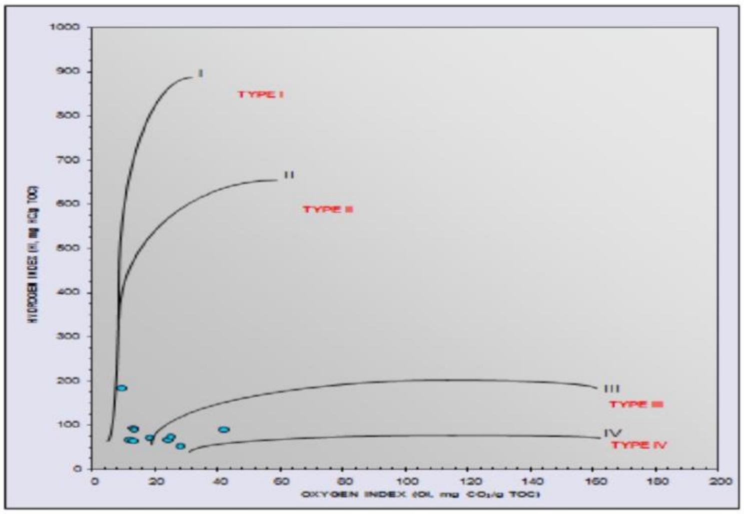

Figure 10. Evolution plot of HI versus OI

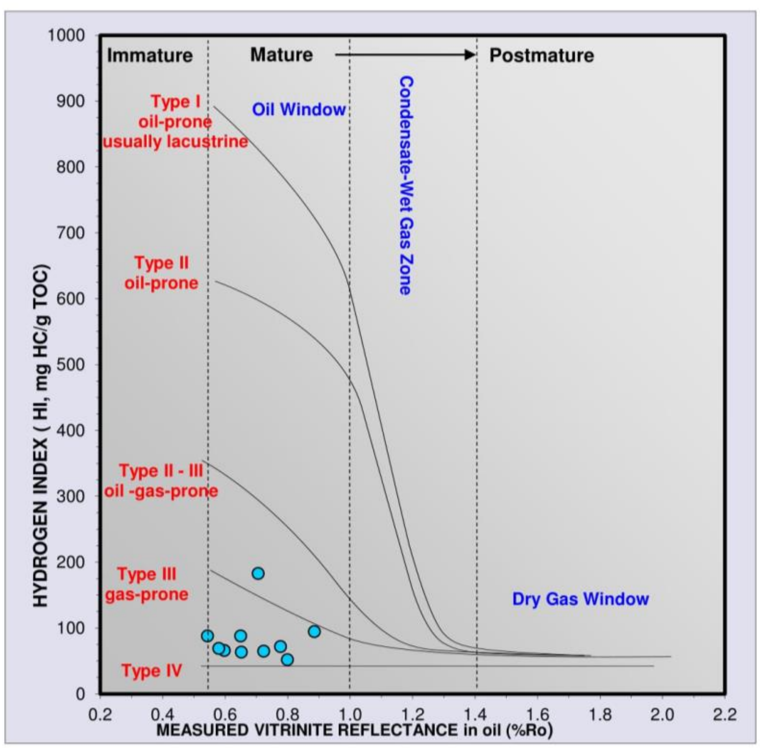

Figure 11. Plot of HI vs calc. % R_o

4.4. Palaeo-temperature

The vitrinite reflectance (%R_o) was used to estimate maximum burial temperatures (T_{burial}) for the Ekenkpon Shale using Barker equation [25]:

$$\ln (R_0) = 0.0096 (T_{burial}) - 1.4$$

where R_0 is vitrinite reflectance(%R_o); T_{burial} is maximum burial temperature (°C). Potential errors ±30°C [24].

Based on the above equation, the T_{burial} for the shale in Ekenkpon Formation range from 82°C to 133°C (mean=106°C) suggesting oil generative window [21-22] (Table 3 and Figure 14).

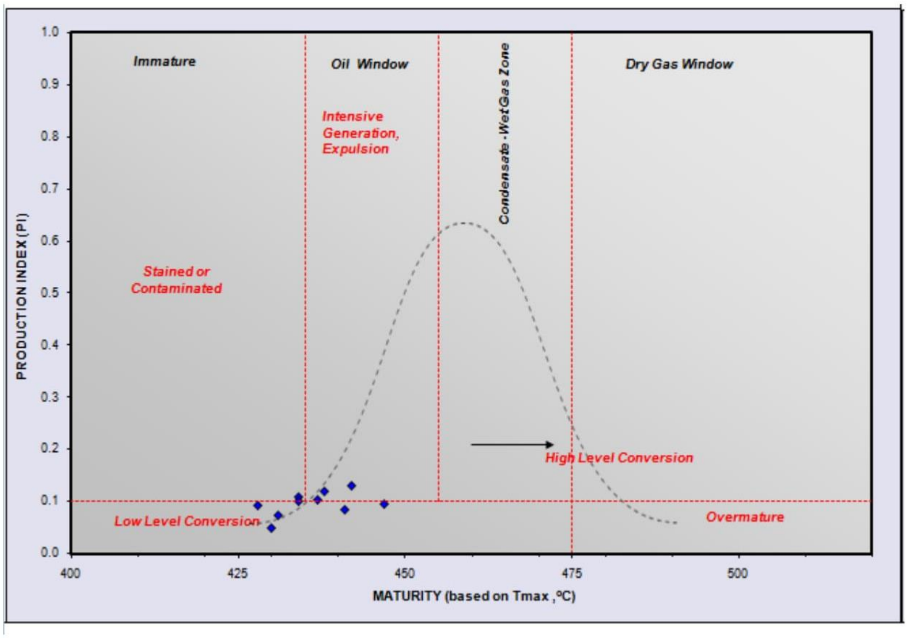

Figure 12. Plot of PI versus T_{max}

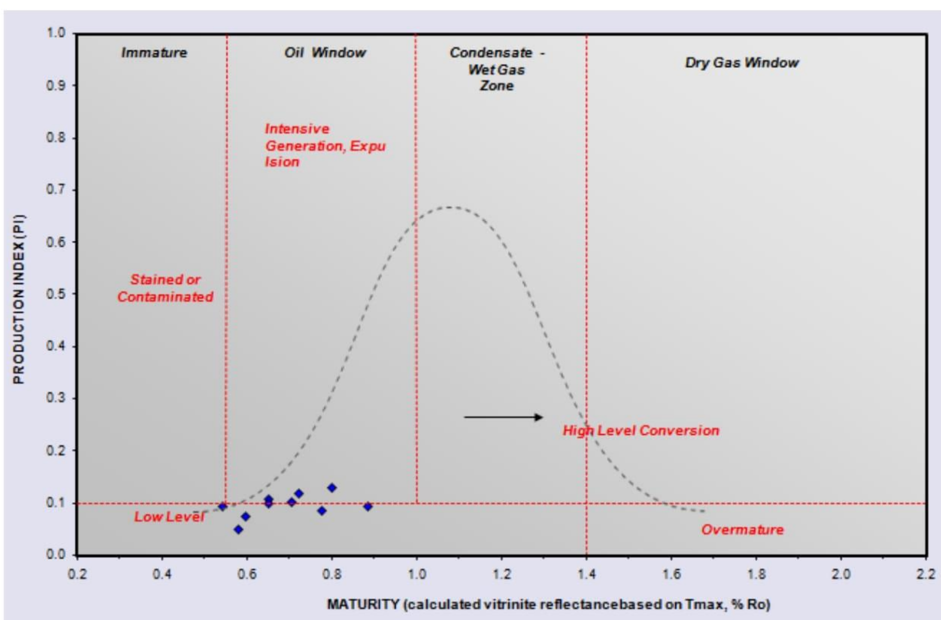

Figure 13. Plot of PI versus calc. %R_o

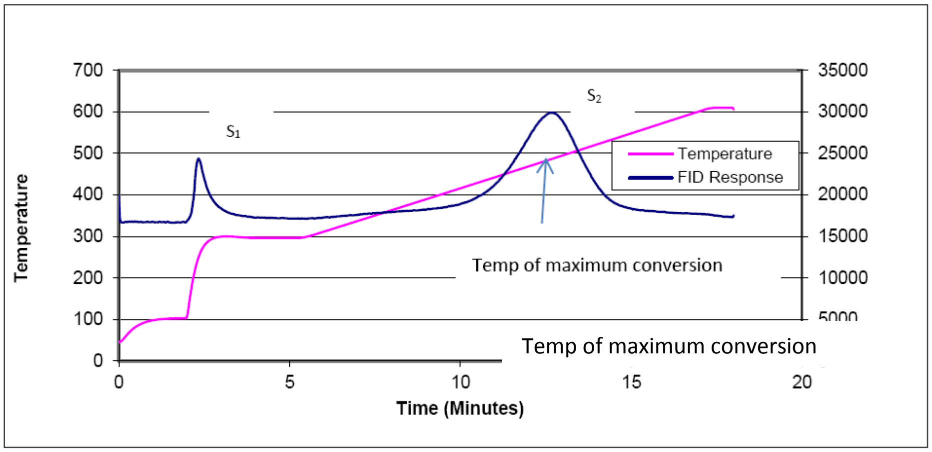

Figure 14. Pyrogram of sample D8 (S1 and S2 peaks identified)

5. Conclusions

Geochemical analyses were carried out on selected samples from the Calabar Flank and the results show that the shale of the Ekenkpon formation were derived from organic matter of terrestrial origin deposited in oxic and/or sub-oxic environment. The geochemical indices show that the shales are immature to marginally mature and the organic matter is predominantly gas prone Type III kerogen.

The level of thermal maturation derived show that the shale sediments are partly within the oil window. This is corroborated with the burial temperature (T_{burial}) value of 106°C suggesting that the shales are within the oil generative window. It is however evident that this study on subsurface samples does not show any significant disparity from the earlier study on surface samples.

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