Organic Matter Quantity, Quality and Maturity Studies of the Paleocene Ewekoro Formation, Southwestern Nigeria

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Abstract

Organic matter quantity, quality and maturity studies was carried out on the Paleocene Ewekoro Formation to assess its source rock potentiality using organic geochemical and petrographic techniques. Organic richness assessment based on Total Organic Carbon content (TOC), Genetic Potential, Soluble Organic Matter (SOM), hydrocarbons generated before pyrolysis (S_1) and residual petroleum potential (S_2) indicates that the carbonate facies of the Paleocene Ewekoro Formation are organic matter lean and has very poor hydrocarbon generation potential. Type of Organic Matter evaluation using hydrogen and oxygen indices, plot of HI against T_{max} and S_2/S_3 ratio shows that they are predominantly kerogen type IV and some type III; of terrestrial organic matter, a characteristic of oxic depositional milieu. Maturity studies presents their status as immature or over mature with low level of convertibility to hydrocarbons. S_2-TOC cross-plot indicates that the organic matter is composed mainly of 'dead' organic carbon with no potential for hydrocarbon generation.

Keywords: Organic matter, kerogen, source rock, hydrocarbon

1. Introduction

Organic geochemical modelling is applicable in the study of origin, migration, generation, accumulation, and alteration of petroleum (Tissot & Welte, 1984). They defined petroleum source rocks as rocks that are capable of generating petroleum given the right maturity. The depositional controls on organic carbon accumulation in carbonate systems are similar to clastic systems, but differs in that carbonate systems can create the physiogeographic restriction necessary for the development of anoxia and enhanced preservation of organic matter by their response to rapid relative sea-level rise. The search for crude oil and natural gas is a costly and risky enterprise, after finding most of the easily detectable reservoirs in the readily accessible areas, hydrocarbon exploration is now proceeding into frontier areas. The Benin basin in southwestern Nigeria is one of the frontier areas that has the potential for hydrocarbon production. Many of the world's most prolific source rocks are developed in marine carbonate depositional systems. Although they constitute a small percentage of all carbonate rocks, organic-rich, fine-grained carbonate rocks are widespread in both time and space and are the probable source of 30-40% or more of the petroleum reserves of the world (Jones, 1984). The Ewekoro limestone in Ogun State, southwestern Nigeria is used as raw material for cement production, this present inquisition assesses the organic facies and source rock potential of the carbonate facies of the Ewekoro formation.

2. General geology

The evolution of the Benin basin is generally believed to be due to rifting phenomenon associated with the separation of the mega-continent called Gondwanaland. According to (Agagu, 1985), the stratigraphy in most parts of the basin is dominated by alternating sands and shales with minor proportion of limestone. The Ewekoro Formation is one of the formations deposited in the Benin Basin (also known as Dahomey Basin or Dahomey Embayment) and assigned Paleocene age based on flora and fauna. The Ewekoro Formation consists of thick fossiliferous limestone. Adegoke (1977), described the formation as consisting of shaly limestone about 13m thick which tends to be sandy and divided into three microfacies.

Detailed stratigraphy of the basin has been delineated by several workers (Jones & Hockey, 1964; Adegoke, 1969; Ogbe, 1970; Billman, 1976; Ako et al., 1980; Omatsola and Adegoke, 1981; Okosun, 1990). However, there is still no unified stratigraphic system for the basin; there are still geological polemics about age and lithological classifications.

Table 1 shows the lithostratigraphy from the oldest to the youngest according to (Jones & Hockey, 1964; Omatsola and Adegoke, 1981; Agagu, 1985): the Cretaceous Abeokuta Group—made up of the Ise Formation, Afowo Formation and Araromi Formation. Shallow marine Paleocene Ewekoro Formation deposited during transgressive episode overlies the Abeokuta Group. The Ewekoro Formation is widespread covering a distance of about 32km from Ghana, eastwards towards the eastern margin of the basin. It finally becomes fine grained into the dominantly

Table1. Stratigraphy of the Benin Basin

PERIOD	**Jones & Hockey (1964)**		**Adegoke & Omatsola (1981)**		**Agagu (1995)**	
	Age	Formation	Age	Formation	Age	Formation
Quaternary	Recent	Alluvium			Recent	Alluvium
Tertiary	Pleistocene – Oligocene	Coastal Plain Sand	Pleistocene – Oligocene	Coastal Plain Sand	Pleistocene – Oligocene	Coastal Plain Sand
	Eocene	Ilaro sandstone	Eocene	Ilaro sandstone/Oshosun shales	Eocene	Ilaro sandstone/Oshosun shales
	Paleocene	Ewekoro Limestone	Paleocene	Akimbo shale/Ewekoro Limestone	Paleocene	Akimbo shale/Ewekoro Limestone
Late Cretaceous	Late Senonian	Abeokuta Formation	Maastrichtian to Neocomian	Araromi shale	Maastrichtian to Neocomian	Araromi shale
				Afowo sandstone/shale		Afowo sandstone/shale
				Ise sandstone		Ise sandstone
Precambrian Basement Complex						

shelly Araromi formation which pinches out against the Western flank of Okitipupa ridge (Adegoke, 1969; 1977). The Akinbo Formation (Paleocene-Eocene) overlies the Ewekoro Formation. The Eocene Oshosun Formation overlies the Akinbo Formation and Oshosun Formation is overlain by the regressive arenaceous Ilaro Formation. Figure 1 is a geological map of Ogun State showing the extent of the Ewekoro Formation and locations where samples were collected.

3. Materials and methods

Limestone samples were collected from outcrops from Ewekoro, Shagamu and Ibese quarries. The samples were taken for organic geochemical and petrographic analysis, to evaluate organic matter richness, maturity, type and facies.

3.1 Total organic carbon content

The total organic carbon contents of the rock samples were determined on powdered samples, using LECO CS – 244 analyzer. About 100mg of the powdered sample was weighed in a special porous ceramic crucible and placed on a cold sand bath. The weighed sample was then wetted with few drops of ethanol (to avoid sporadic reactions) and treated with some drops of 10% diluted HCl until no further reactions occurred. The sample was left on the sand bath for 12 hours at 80^0C in order to evaporate the excess water. Thereafter, the sample was transferred into a laboratory vacuum oven where it was kept for another 12 hours at the temperature of 50^0C. Before bringing the sample in the combustion chamber ca. 1g of copper turnings was added as catalyst. The sample was then burnt in the LECO CS-244 analyzer, in the presence of oxygen at a temperature of about 1300^0C. The evolved gas was measured quantitatively and simultaneously by infrared detectors and recorded as percent carbon. Before the samples were analyzed, the instrument was calibrated with standards of known percent carbon.

3.2 Rock-Eval-Pyrolysis

According to Espitalie *et al.,* (1977), Rock-Eval Pyrolysis offers a rapid method for the characterization of kerogen types and for the determination of the maturity of organic matter. The measurement was carried out with the Rock-Eval II device of Delsi Instrument. The weight of the sample to be evaluated was based on the total organic carbon content of the sample. The samples were weighed in a metal crucible that was previously burnt out at a temperature of 600^0C under nitrogen atmosphere. The crucible and the content were then transported automatically into the oven room and flushed for 3 minutes with helium to remove oxygen and CO_2. Subsequently the samples were placed in the oven for 3 minutes and heated to a temperature of 300^0C. During this assay, free and absorptive hydrocarbon and hydrocarbon-like compounds were volatilized and quantitatively determined with the help of flame ionization detector (FID). This was then registered as the S_1 – peak.

The oven temperature was again increased to 550^0C at a programmed rate of 25^0C/minute and held constant for 1 minute. At this second temperature phase (300-550^0C) the solid organic matter was pyrolyzed and the mobilized hydrocarbon and hydrocarbon like compounds were measured simultaneously and quantitatively with FID as S_2-peak. The CO_2 produced during the temperature interval (300-390^0C) was registered with the thermal conductivity detector TCD as the S_3-peak. The mobilized products of S_1 and S_2-peaks could be normalized to the weight of rock (mgHC/g rock) and to the total organic carbon (mgHC/g TOC). T_{max}-value corresponds to the temperature at which the pyrolytic yield of hydrocarbon (S_2-peak) reaches its maximum. The T_{max} – value increases with increasing maturity (Espitalie *et al.,* 1985).

3.3 Vitrinite Reflectance

The sample was crushed and milled after which the sample was placed in a 50- mL polycarbonate test tube for kerogen concentration. ZnBr2 was added to each tube to wet the sample and stirred with a stirring motor and paddle. Thin slurries were centrifuged to separate organic from inorganic material. The kerogen concentrate was then cast in epoxy on standard petrographic slides. Qualitative microscopic analysis of the organic materials were carried out using reflected light microscope equipped with white incident light and blue light irradiation. The reflectance was measured on polished sections by means of a photometer mounted on a microscope. The VR measurements were carried out with oil objectives 320 λ and 40/0.85 interference filter (54 nm).

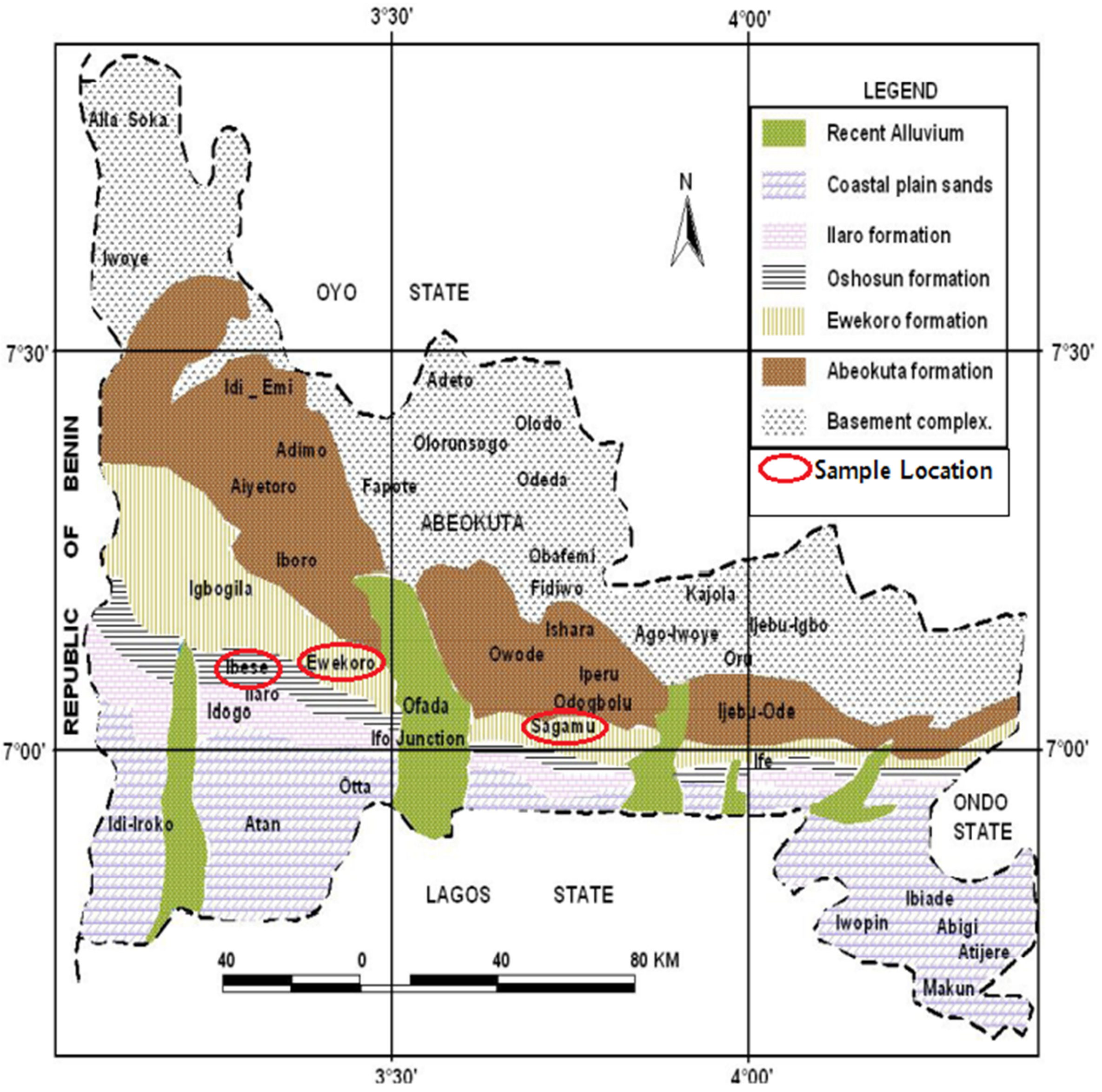

Figure 1. Geological map of Ogun State showing the extent of the Ewekoro Formation and locations where samples were collected.

4. Results and discussion

4.1 Organic richness (organic quantity)

Organic richness is the ability of hydrocarbons to be generated (Peter and Moldowan, 1993). Since petroleum is a generative product of OM disseminated in the source rock, the quantity of the petroleum should be correlatable with the organic richness of the potential source rock (Dow, 1977; Welte et al., 1981; Tissot & Welte, 1984). Parameters used in this work to determine OM richness are: total organic carbon content (TOC), genetic potential, hydrocarbons generated before pyrolysis (S_1) and residual petroleum potential (S_2), and Soluble Organic Matter (SOM). Table 2 shows the results of organic geochemical and petrographic analysis of the Ewekoro limestone.

4.1.1 Total Organic Matter content (TOC)

It is known that adequate amount of organic matter, measured as percentage total organic carbon (TOC), is a necessary pre-requisite for sediment to generate oil & gas (Cornford, 1986). The TOC is the weight percent of carbon in the source rock and is measured as the amount of carbon dioxide produced when acid-leached rock sample is combusted in the presence of oxygen. Average carbonate source rocks have TOC of 0.7% (Tissot, & Welte, 1984). The studied Ewekoro limestone have TOC values between 0.04 and 0.23 (Av. 0.16), which indicates that they are not rich in organic matter and have poor petroleum potential. According to (Leythaeuser, 1973; Reed, 1977; Clayton & Swetland, 1978), organic matter in outcrops that had previously been buried to great depths, do undergo alterations because of sub-aerial exposure. Based on the study of the Upper Cretaceous shales of Utah (Leythaeuser, 1973) concluded that there was 25% loss of TOC and 50% loss of soluble organic matter with sub aerial weathering. Clayton & Swetland (1978), also showed that in the Permian Phosphoria Formation, there was a 60% loss in TOC and 50% loss in the C_{15} hydrocarbon at depths $\leq$ 0.79m, but such effects were hardly noticed below depths of 0.79m. Also large sedimentary basins may have different geothermal heat flows in certain parts of the basin; timing of oil generation and the composition of the products may vary even if the source rock is the same all over the basin (Rùllkőtter, 1992). Though for this present inquisition, fresh samples were sought for as much as possible, but based on the aforementioned observations, it is possible that the OM values of the rocks studied may be higher if 100% fresh samples were utilized.

Table 2. Results of organic geochemical and petrographic analysis

Sample ID	TOC	S_1	S_2	S_3	S_2/S_3	S_1/TOC*100	Tmax (°C)	Meas. % Ro	HI	OI	PI	GP
Ew I	0.05	0.03	0.09	0.04	2	60	435	0.24	180	80	0.25	0.12
Ew II	0.05	0.04	0.09	0.06	2	80	437	0.23	180	120	0.31	0.13
IBEI	0.05	0.01	0.00	0.04	0	20	420	0.22	0	80	1.00	0.01
IBEII	0.04	0.01	0.00	0.06	0	25	419	0.25	0	150	1.00	0.01
AK-Sh 1	0.22	0.01	0.00	0.05	0	5	425	0.17	0	23	1.00	0.01
AK-Sh 2	0.20	0.01	0.00	0.04	0	5	423	0.17	0	20	1.00	0.01
AK-Sh 3	0.22	0.01	0.00	0.06	0	5	430	0.15	0	27	1.00	0.01
AK-Sh 4	0.20	0.01	0.00	0.04	0	5	420	0.13	0	20	1.00	0.01
SHA I	0.22	0.01	0.00	0.06	0	5	418		0	27	1.00	0.01
SHA II	0.20	0.01	0.00	0.06	0	5	422		0	30	1.00	0.01
SHA III	0.21	0.01	0.00	0.05	0	5	425		0	24	1.00	0.01
SHA IV	0.23	0.01	0.00	0.05	0	4	423		0	22	1.00	0.01

4.1.2 Hydrocarbons generated before pyrolysis (S_1)

This is the amount of hydrocarbon in the source rock already generated by geospheric maturity and merely distilled out of the rock during pyrolysis. The value of S_1 could be used to measure the source rock generative potential (Peters, 1986). S_1 minimum value for good source rocks is 1.0 mgHC/g dry rock (Pennsylvania State geology survey, 2011). The S_1 values range from 0.01 and 0.04 (Av. 0.01), which indicates that they have poor generative potential (Peters and Cassa, 1994).

4.1.3 Residual Petroleum Potential (S_2)

S_2 represents the bitumen that would be generated if burial and maturation continued to completion and decreases with increasing maturation (Leckie et al., 1988). The S_2 corresponds to the hydrocarbons that would evolve from the sample during the second programmed heating stage of pyrolysis. These hydrocarbons result from cracking heavy hydrocarbon and from the thermal breakdown of kerogen (McCarthy et al., 2011). S_2 minimum value for good source rocks is 5.0 mgHC/ dry rock (Pennsylvania State geology survey, 2011). S_2 values for the samples range from 0 and 0.09 (Av. 0.02)—they have poor generative potential (Peters and Cassa, 1994).

4.1.4 Genetic Potential (Pyrolytic or Potential Yield)

Genetic Potential is the summation of free hydrocarbon and hydrocarbon generation through thermal cracking (Peter & Moldowan, 1993). It is derived from Rock Eval pyrolysis from the summation of S_1 and S_2 values. According to Dymann *et al.,* (1996) a good source rock should have a GP of 2kgHc / ton of rock. GP values for the Ewekoro limestone range from 0.01 and 0.13 (Av. 0.03). Classification based on Genetic Potential by Tissot & Welte (1984), suggests no hydrocarbon source rock potential. A cross plot of S_2 versus TOC, (Figure 2) also indicates that the limestones are OM lean.

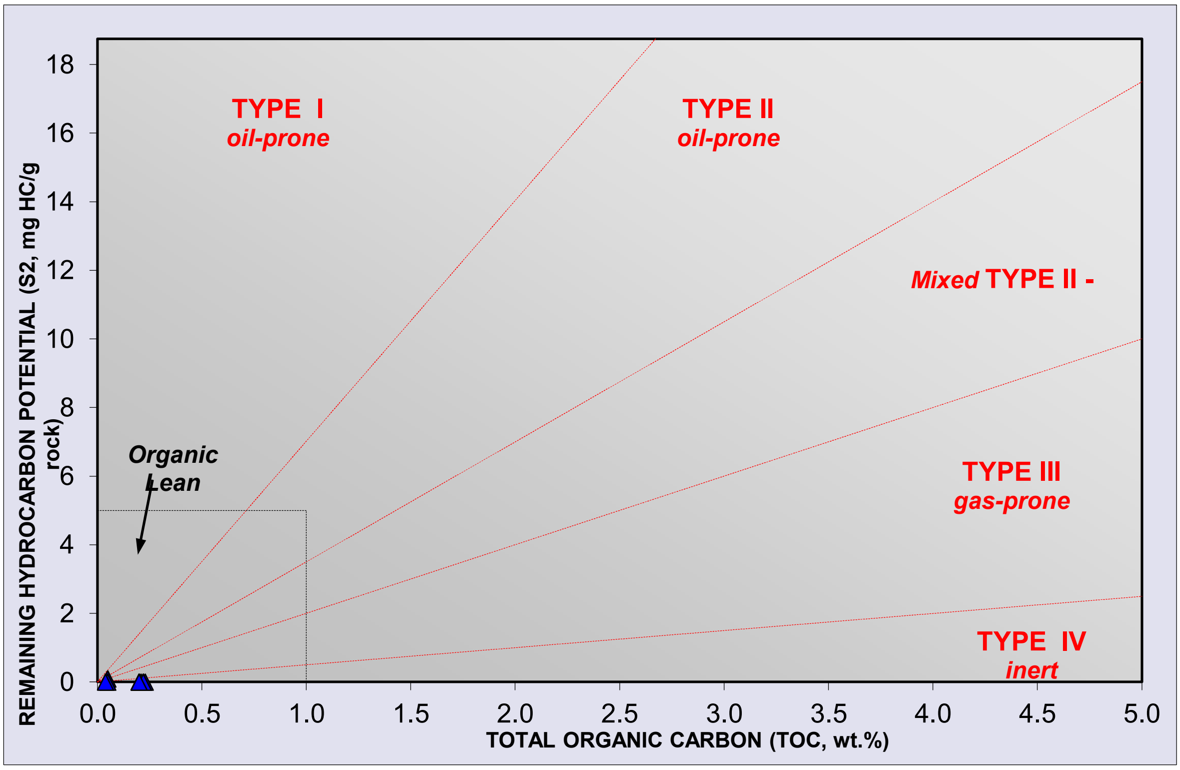

Figure 2. Kerogen quality

4.1.5 Soluble Organic Matter (SOM)

Soluble organic matter is used to determine the source rock potential, maturity and depositional environment. The significant of this is that extraction and the determination of yield of SOM allow for identification of hydrocarbon-rich sediment (Peter and Cassa, 1994). The SOM values range between 360 – 420 ppm (average 385 ppm). This shows that the limestone has a poor petroleum generation potential (Peter and Cassa, 1994).

4.2 Type of Organic Matter (Organic Matter Quality)

Many organisms contribute to the OM present in petroleum source rocks. Since these organisms differ in their contents of lignin, carbohydrates, lipids, protein, resin, etc., the preserved organic matter exhibits parallel diversity. Sedimentary, diagenetic, catagenetic and metagenetic processes modifies the original constituents further. The diversity influences the convertibility of kerogen to petroleum: some being oil-prone or gas-prone, while others are not convertible to petroleum (Dow, 1977). Quality organic materials must be present for a geologic unit to be considered a potential source rock (Pennsylvania State geology survey, 2011). The parameters used here for the evaluation of the type of OM are: hydrogen and oxygen indices (HI & OI), plot of HI against T_{max} and S_2/S_3 ratio

4.2.1 Hydrogen and oxygen indices

Hydrogen index (HI) represents the hydrogen richness and oxygen index (OI) depicts the oxygen content of the kerogen, both relative to the total organic carbon content (Snowdon, 1989). HI values for range from 0 and 180 mgHC/g TOC (Av. 30). Oxygen index values ranges between 20 – 150 $mgCo_2/g$ TOC, with an average of 52 $mgCo_2/g$ TOC. McCarthy et al., (2011) classified source rocks with HI between 50 and 200 as gas-prone. According to McCarthy et al. (2011), the null values (HI) for the Ewekoro limestones (except for two samples) shows that the type of OM present (Type IV) cannot generate hydrocarbon. Figures 3 and 4 shows that they contain kerogen type IV which indicates an oxic depositional milieu.

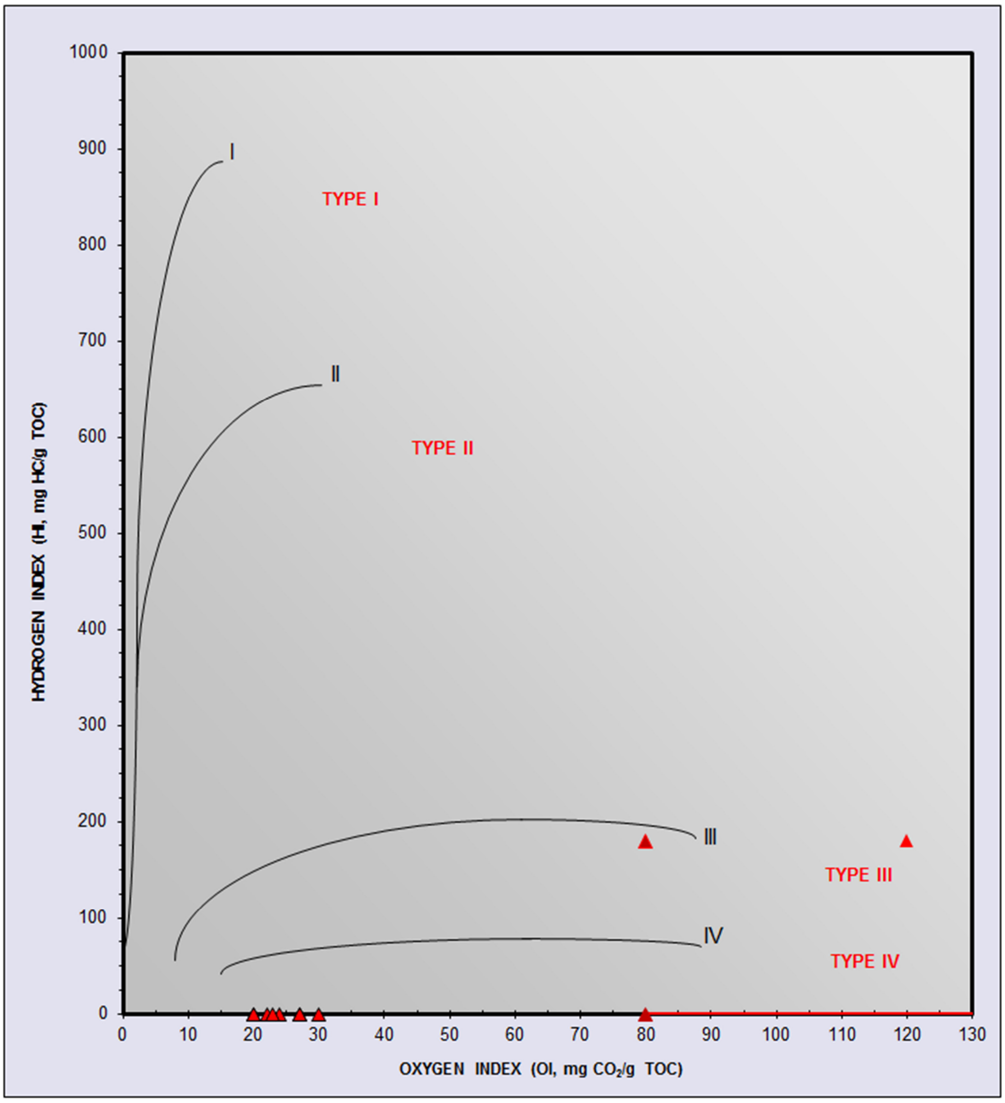

Figure 3. Type of Kerogen

4.2.2 S_2/ S_3 Ratio

This ratio is theoretically equivalent to the ratio HI/OI and useful for describing the kerogen type and the type of hydrocarbon product likely to be expelled by a rock (Peters, 1986; Leckie, et al., 1988; Peters & Cassa, 1994). S_2/S_3 ratio for the limestones range from 0 to 2 (Av. 0.3), which is of Type IV OM—it is inert and cannot generate hydrocarbon (Weber and Green, 1981; Peters, 1986; Peters & Cassa, 1994).

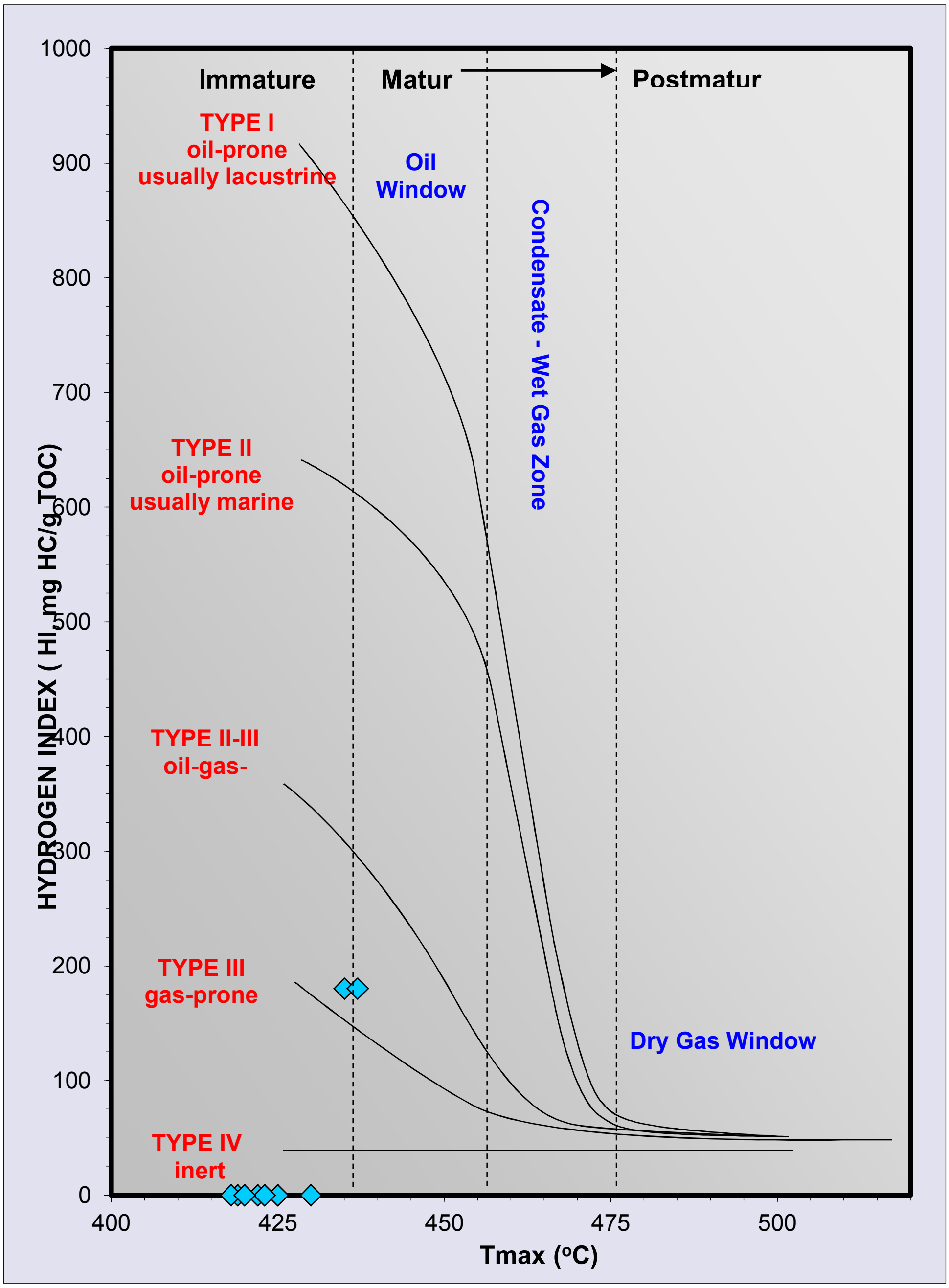

Figure 4. Type of Kerogen and Maturity

4.3 Organic Matter Maturity

Maturation is the process of chemical change in sedimentary organic matter, induced by burial, i.e. the action of increasing temperature and pressure over geological time (Miles, 1989). Heroux, et al. (1979) presented a comprehensive review of the non-biomarker geochemical parameters used for the determination of the maturity level of organic matter. The maturation parameters employed for this study include: Production Index (PI), T_{max}, S_1/TOC ratio, Vitrinite Reflectance (VR_o) and the HI & OI indices.

4.3.1 Production index (PI)

The PI is a maturation parameter derived from pyrolysis and it is the ratio of already generated hydrocarbons to potential hydrocarbon (Miles, 1989). It is calculated from the ratio S_1/S_1+S_2. It increases with maturity and also indicates the presence of epigenetic hydrocarbons. Colloquially its upper and lower limits indicates the oil “birth” and “death” (Hunt. 1979; Tissot & Welte, 1984; Peters, 1986) or the early mature to late mature boundaries respectively (Peters and Cassa, 1994), provided there is no migration into or out of the sample under investigation (Leckie, et al., 1988). According to Dembicki et al., (1983); Epistalie et al., (1980), and Horsfield & Douglas (1980), the PI is affected by mineral matrix, and also by migration (Clementz, 1979). The PI values for Ewekoro samples ranges between 0.25 – 1 (Av. 0.88). These values correspond to peak to late mature stage (Miles, 1989; Peters and Cassa, 1994), meaning that they attained thermal maturity for oil but no oil was generated, which may be due to the type (two types of organic facies) and quantity of organic matter.

4.3.2 S_1/TOC ratio

The S_1/TOC ratio (bitumen content) is the Normalized Oil Content and significant as an indicator of the approach of the “oil window” and in conjunction with the Production Index (PI), enables the recognition of the presence of migrated hydrocarbons (Babaie *et al.,* 1992). S_1/TOC ratios is between 4 and 80, with an average of 18.7. Only 2 samples have values >50 which corresponds to mature tight source rock (Jarvie and Tobey, 1999; Jarvie and Baker, 1984), while the rest have ratios <50 indicating lean source rock (Jarvie and Baker, 1984); and low or overmature source rock (Ruble, et al.). A cross plot of S1 versus TOC (Figure 5) reveals that whatever hydrocarbon that might be present in the rock samples were indigenous without migration from outside source.

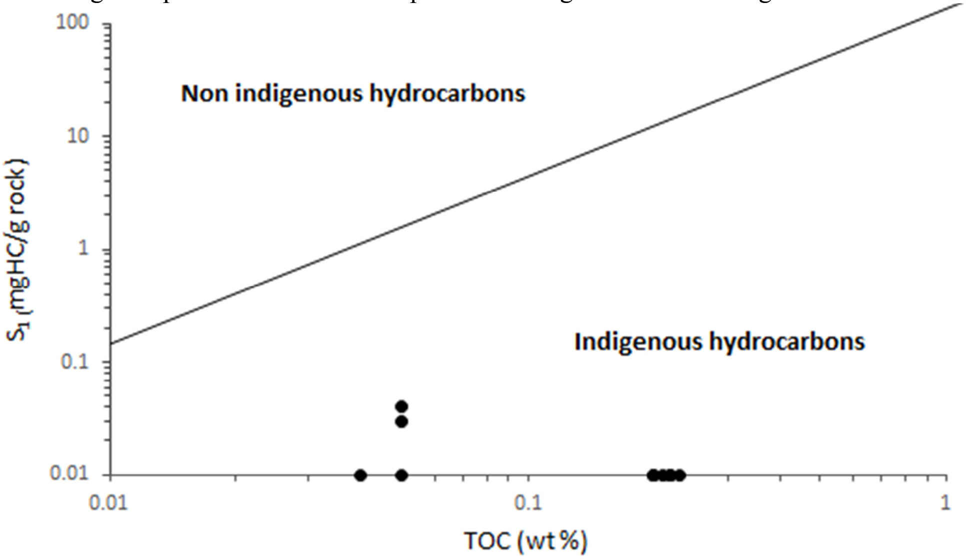

Figure 5. Plot of S1 versus TOC illustrating the indigenous nature of potential hydrocarbons in the Ewekoro Formation. Modified from Al-Ameri, et al., (2011).

4.3.3 T_{max}

This the temperature at which the pyrolytic yield of hydrocarbons from a rock sample reaches its maximum, using Rock Eval pyrolysis and it is kerogen dependent. It is affected by mineral matrix (Epistalie et al.,1980; Horsfield & Douglas, 1980); organic contamination (Peters, 1986); and migration (Clementz, 1979). Acording to to Snowdon, (1995), the observed T_{max} values from Rock Eval pyrolysis may be anomalous (depressed). The lowest and highest T_{max} values are 418 and 437 °C (Av. 425), which indicates that all the samples are thermally immature (Peters and Cassa, 1994) except for one sample with T_{max} value of 437 0C, which might be due to the presence of type III organofacies. A plot of Hydrogen Index against T_{max} (Figure 4) shows that the kerogens are of Type IV and immature.

4.3.4 Vitrinite Reflectance (VR_o)

It is a measure of the amount of light reflected by vitrinite present in the rocks organic component, it is used as maturity indicator but dependent on kerogen type (Peter and Moldowan 1993). This parameter is based on the change in the reflectance of polished vitrinite particles with increasing time and temperature. Increase in reflectance are caused by the progressive aromatization of the kerogen with accompanying loss of hydrogen in the form of hydrocarbon gases; the end product of the process is graphite (Miles, 1989). VR_O values range from 0.13-0.25 %, (Av. 0.20)—they are thermally immature and a plot of Hydrogen Index (HI) against Vitrinite Reflectance (Figure 6) also shows that the samples are immature. Also, figures 7 and 8 shows that the kerogen are immature and has low level of convertibility to hydrocarbons.

4.3.5 Hydrogen (HI) and Oxygen indices (OI)

The Hydrogen Index (HI) can be used to indicate maturity if the kerogen type is known (Miles, 1989). The Hydrogen Index is between 0 and 180 mg HC/g TOC (Av. 30), while Oxygen index 22 – 150 mg HC/g TOC (Av. 52)—these OI values are less than 200 mg CO2/g rock and indicate that there is no intensive weathering or mineral decomposition in analyzed rock samples (Jarvie and Tobey, 1999). According to Miles (1989), immature Type III kerogen has HI ranges between 0-300 mg HC/TOC and OI values greater than fifty (>50) mgCo2/g TOC are characteristics of immature kerogen. Using the HI and OI parameters, the limestone samples are immature, although some of the OI values are below 50, this status could be either Late or post mature.

5. S_2-TOC cross-plot

Oil and gas potential can also be determined using the method of Dahl et al. (2003). This approach uses Rock-Eval and TOC data in a simple S_2-TOC cross-plot to determine the oil and gas potential of a specific source rock section (Justwan & Dahl, 2005). The slope of the regression line represents the average hydrogen index of the sample population. Often, the regression line does not intersect the origin and the intersection with the TOC-axis represents the 'dead' organic carbon (Cornford 1998). Figure 9 shows that regression line did not intercept the origin but intercepts the TOC-axis, which represents type IV inert kerogens with no hydrocarbon potential.

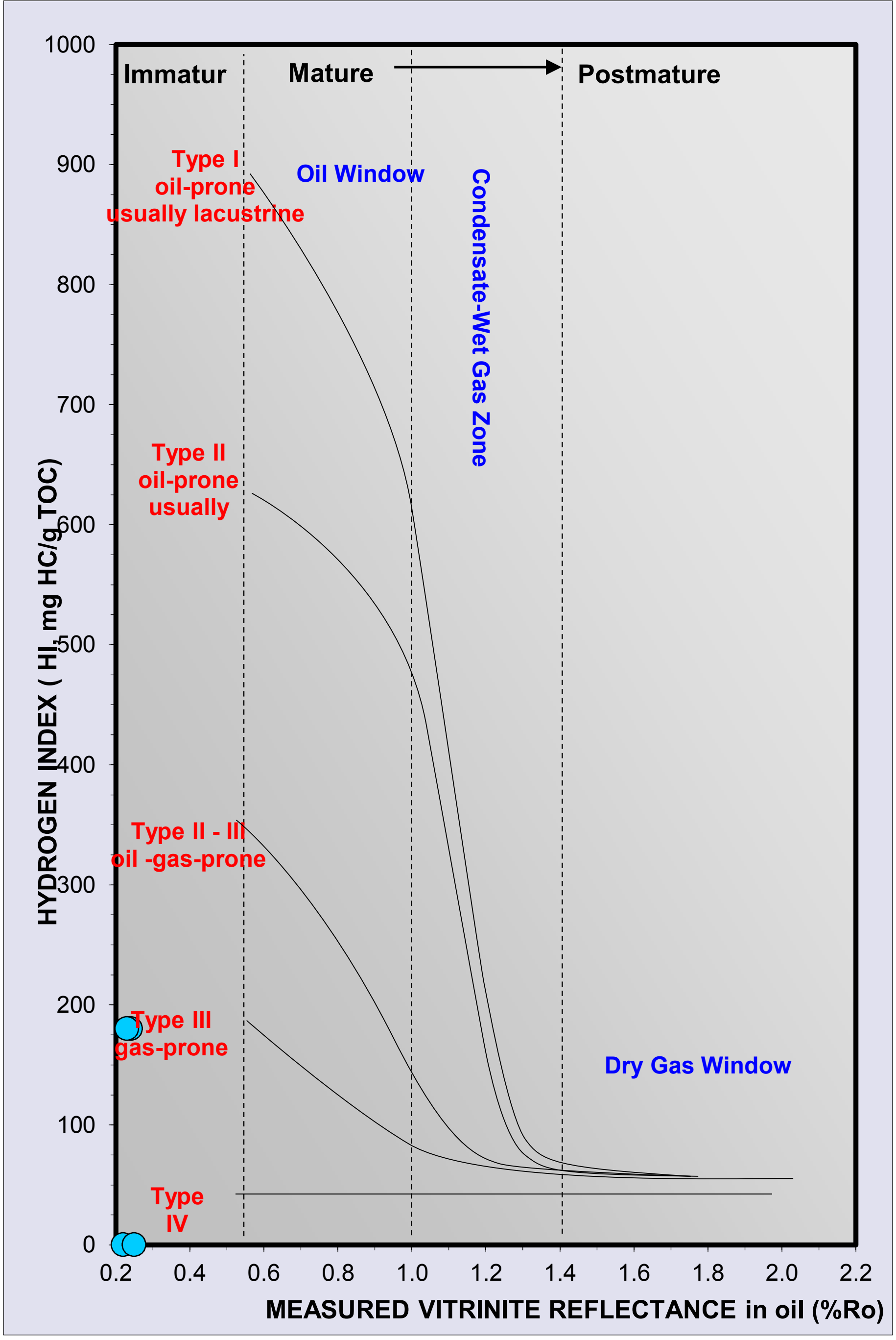

Figure 6. Kerogen Maturity based on Vitrinite Reflectance

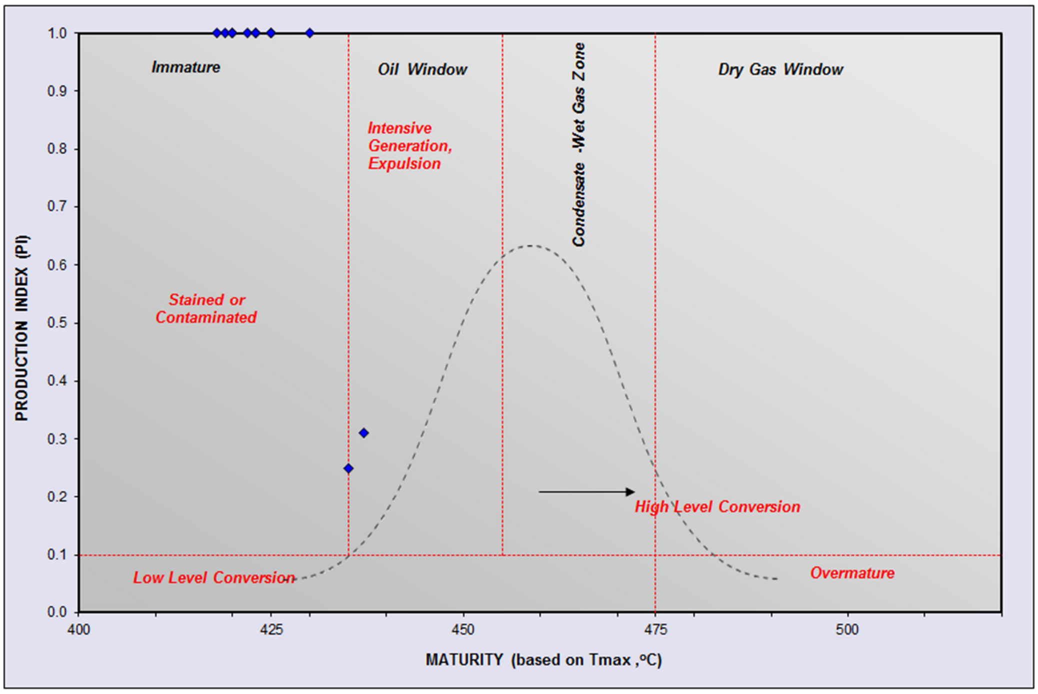

Figure 7. Kerogen Maturity based on PI and T_{max}

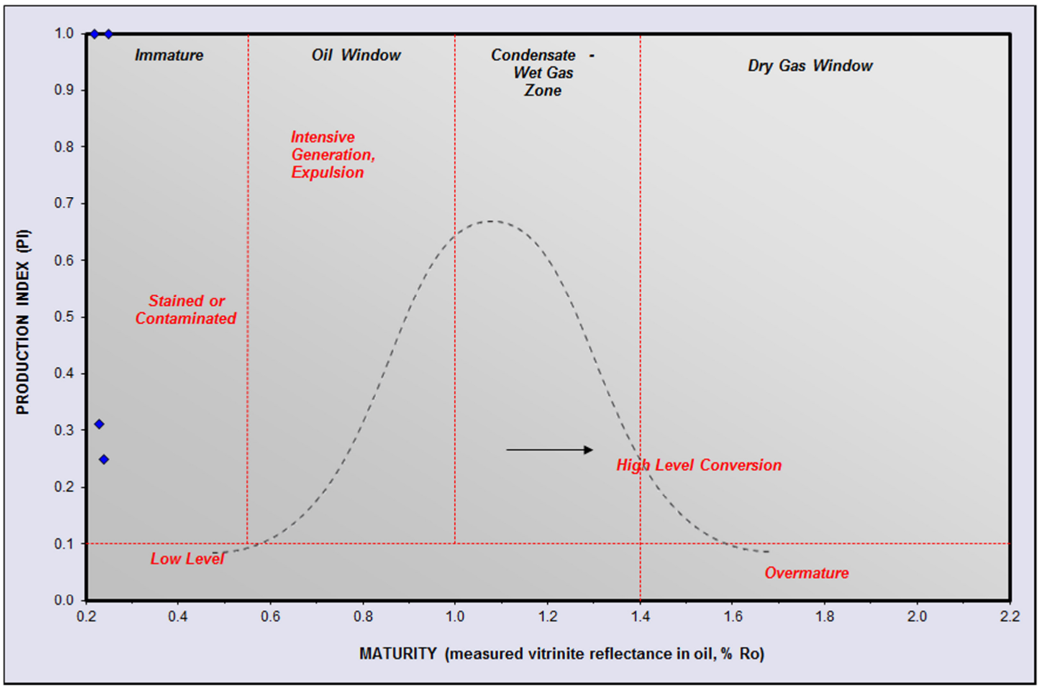

Figure 8. Kerogen Maturity based on PI and Vitrinite Reflectance

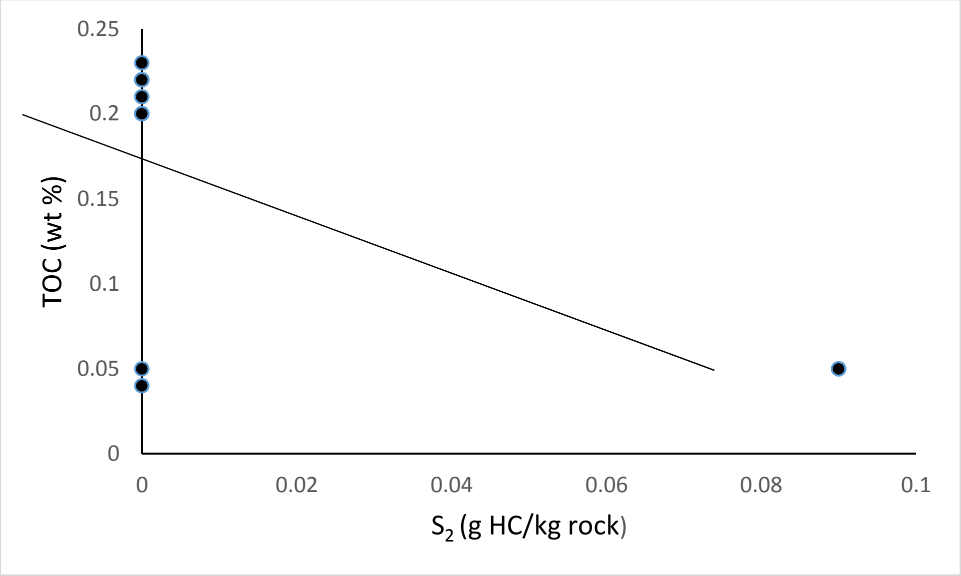

Figure 9. S_2-TOC cross-plot

6. Conclusions

Organic richness assessment indicates that the Paleocene Ewekoro limestone are organic matter lean and has very poor hydrocarbon generation potential. Type of Organic Matter (Organic Matter Quality) evaluation shows that they are predominantly kerogen type IV and some type III terrestrial organic matter, a characteristic of oxic depositional milieu. Maturity studies on the kerogens presents their status as immature or overmature with low level of convertibility to hydrocarbons. S_2-TOC cross-plot indicates that the OM is composed mainly of 'dead' organic carbon with no potential for hydrocarbon generation. Thus, the Paleocene Ewekoro Formation is not a good carbonate source rock.

Acknowledgement

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