COMPARATIVE EVALUATION OF HYDROCARBON POTENTIAL OF NKPORO AND ENUGU SHALE DEPOSITS IN OKIGWE-ENUGU AXIS, ANAMBRA BASIN SOUTHEASTERN NIGERIA

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ABSTRACT

Four rock samples collected from Nkporo and Enugu Shales (Leru, 11369 and Ivo River, 11368) and (Emene, 11367 and New Mkt, 11366) respectively, Okigwe-Enugu axis Anambra basin southeastern Nigeria were subjected to geochemical analysis for comparative evaluation of hydrocarbon potentials of the both shales. This is to determine their quantity, quality and thermal maturation status and compare their lateral equivalent. The Total Organic Carbon (TOC) analysis was performed by means of the LECO CS125 Carbon Analyser machine module and the Rock Eval pyrolysis was performed using the Rock-Eval LECO 6 machine module. The result revealed that the Total Organic Content (TOC) of shale samples recovered from Nkporo Shale ranged from 2.05 to 4.77wt%, and Enugu Shale samples with TOC values of 2.41 to 2.69wt%. These values essentially exceeded the threshold value of TOC ≥ 0.5wt%, which suggested that the shales are from a good to very good source rocks. The Hydrogen Index (HI) values of sediments from Nkporo Shale are 46 to 480mgHC/g TOC with an average value of 263mgHC/g TOC and those from Enugu Shale have the HI values of 50 to 160 mgHC/g TOC with an average value of 105 mgHC/g TOC; which shows that the quality of Nkporo and Enugu Shales were characterized by Type IV/II and mixed Type II-III/III kerogens respectively. This is also confirmed in the van krevlin diagram plot of HI/OI and S_2/TOC plots. The inferred environment for the both shale were mix marine and terrestrial environment. The maximum Temperature (T_{max}) for Nkporo Shale ranged from 428°C to 438°C with an average value of 433°C and that of the Enugu Shale ranged from 432°C to 435°C, with an average value of 433.5°C. These temperatures implied that both Nkporo and Enugu Shales range from immature to early mature stage of the source rocks. Thus the maturity indicates that the Nkporo shale 11369 and 11368 were mature to generate dry gas-inert and immature to generate oil respectively. However the Enugu shale samples 11367 and 11366 indicating early mature and immature to generate oil-gas, and gas respectively. The S_2 values obtained in this work suggested that the Nkporo shale has excellent potential to generate oil than gas if thermal maturation continues. And Enugu shale has fair potential to generate gas with little oil if thermal maturation continues. Based on this study TOC, T_{max}, HI, maturation, kerogen type attested that both Shales deposited in the same environmental setting and therefore contain same organic matter.

Keywords: Rock-Eval, Pyrolysis, Shale, Threshold and Nkporo.

1. Introduction

It is necessary, to identify rocks and its contents in source rock evaluation through organic geochemical analysis. Oil and gas are said to be formed when the remains of dead animals and plants incorporate in sediments, buried and formed into rocks when heated depth in the subsurface. The oil and gas then migrate from the source rock to the reservoir rocks to positions of entrapment or accumulation in pools. In this basin of study, oil seepages occur that attracted the interest for this study.

The study area is located in the southeastern part of the Anambra Basin. The study area is located between latitudes 05º55.017[1]N to 06º30.229[1]N and longitudes 007º25.544[1]E to 007º41.178[1]E Anambra Basin (Figure 1). The stratigraphic setting of interbedded shales and sandstones with occasional limestones seen in outcrops and subcrops in the basin aroused the interest of oil companies exploring for Oil in Southeastern Nigeria in the 1940s. Initial efforts were unrewarding and this led to the neglect of the basin in favor of the prolific Niger Delta Basin. The recent policy of the Nigerian Government to encourage active exploration activities for hydrocarbons in marginal oil fields and inland lakes has led to a renewed interest in the Anambra Basin. The Anambra basin is a frontier basin, the hydrocarbon, organic matter content of the basin and the exploration activities in the basin have been discussed by various authors; Ekweozor (2006), Iheanacho (2010), Unomah (1993), Ehinola *et al.* (2005), Akaegbobi (2005) etcetera.

Organic matter present in sedimentary rocks usually represents the lowest proportion of the sedimentary fraction. It is constituted by organic molecules (under the form of monomers and polymers) deriving directly or indirectly from the organic part of the organisms (composed by the elements C - carbon, H - hydrogen, O - oxygen, N - nitrogen, and S - sulfur). Skeletal parts, shells, bones, spines, and teeth are not included (Tissot & Welte). The characterization of the organic matter contained in sediments and sedimentary rocks is an important issue for petroleum geoscientists. The method used depends largely upon the age of the organic matter, the background of the investigators and the objectives of any given study. The integrated use of geochemical techniques is particularly under-utilized (Joao et. al., 2012).

The type of organic matter deposited and incorporated in sediments depends on biological productivity. The chemical composition of the biomass is mainly dependent on the physical and chemical environment of the biological habitat and the evolutionary level of the organisms. Determining environmental factors include light, temperature, nutrients, and conditions of water such as currents, temperature, and etcetera, besides the presence of groups of organisms. Basically all organisms are composed of the same chemical constituents such as proteins, carbohydrates, lipids, and lignins-tannins.

The accumulation of organic matter in sediments is controlled by a limited number of geological conditions. The main factors that control the accumulation of organic matter include the production of the biomass, the degradation processes and transport of the organic matter. The quantity and quality of the organic matter accumulated in sediment are basically the result of the combined influence of the biomass productivity, biochemical degradation and of the organic matter depositional processes. The accumulation is practically restricted to sediment deposited in aquatic environments, which must receive a certain minimum amount of organic matter. In subaerial sediments, organic matter is easily destroyed by chemical or microbial oxidation. This organic matter can be supplied on the form of particles of organic matter or as dissolved organic matter (components) (Joao *et al*., 2012).

Among the liberation of biological products in the adjacent environment and its incorporation to the sediments, occurs the intervention of a number of biological factors, which will influence its chemical structure and will determine its space distribution in the deposits. Organic matter consists of a diverse variety of compounds, which are subjected to microbial attack. The composed biomolecules in living tissues is thermodynamically unstable. When such molecules are secreted or excreted by the organisms, or even after the death of the organism, they tend to lose their integrity and they can be transformed in simple stable components such as CO_2, CH_4, NH_3, H_2S, and H_2O. This process of degradation can be accomplished by the physicochemical processes such as oxidation, photolysis and thermolysis (Joao *et al*., 2012).

The chemistry of organic matter contained within a sedimentary rock changes over time, reflecting its temperature and burial history. This change or thermal maturity is measured and can be combined with quality of organic matter. The maturity level is the product of a number of variables, such as tectonic setting, burial history and thermal history.

From the start of burial, organic matter in sediments suffers numerous compositional changes, which are caused initially by the biological activity (microbial) following by the action of temperature and pressure. This continuous series of processes is denominated by thermal maturation and it is divided into stages of evolution: diagenesis, catagenesis, metagenesis and metamorphism (Joao *et al*., 2012).

Petroleum geochemistry is a branch of geochemistry that studies the application of chemical principles to the study of the origin, generation, migration, accumulation and alteration of petroleum, and the use of this knowledge in exploration and production of oil and gas. The determination of the most favorable petroleum exploration targets depends on the geochemical knowledge of source rocks and on the knowledge of generation, migration and accumulation processes combined with the geophysical and geological features of the sedimentary basin under evaluation (Giovanni, 2002).

The three major features of a source rock: The quantity of organic matter, quality capable of yielding moveable hydrocarbon components are products of the depositional setting and the thermal maturity is a function of the structural and tectonic history of the province (Mohamed et. al., 2014). Source rocks can be divided into four major categories; Potential, Effective, Relic effective and Spent. These features can be identified by using one or combinations of the geochemical analysis such as; TOC Analysis, SOM Analysis, Rock Eval Pyrolysis and etcetera (Mohamed et. al., 2014).

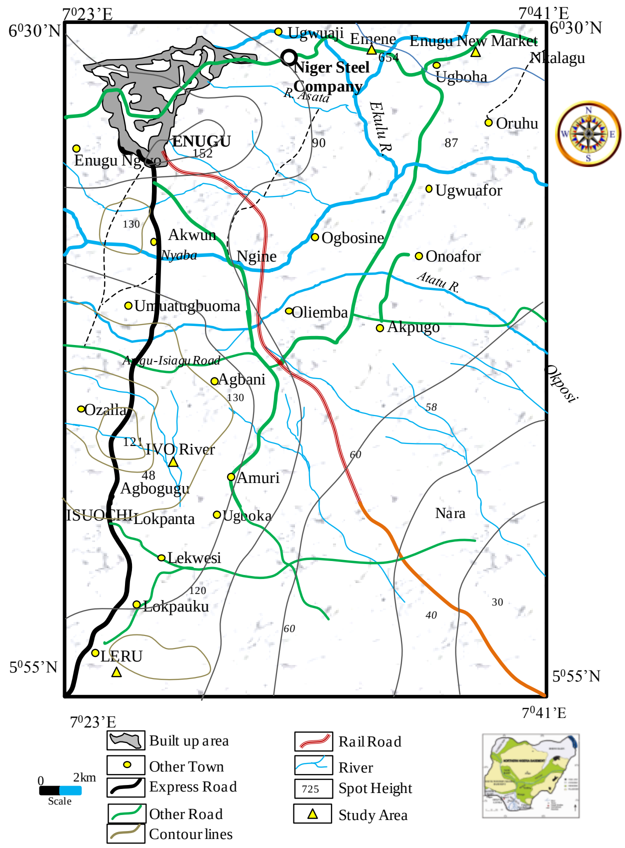


Figure 1: Location and topographic map of the study Area Okigwe-Enugu axis

2. Regional Geology and Stratigraphy

In the Early Cretaceous times, tectonic activities started in the Southern Nigeria, thus, separated African plate from South American and led to the opening of the Atlantic. This resulted in the development of the Benue Trough that stretched in a NE-SW direction and resting unconformably upon the Pre-Cambrian Basement Complex (Salufu et. al, 2015). In the Santonian times, the Southern Benue Trough experienced a tectonic activity that folded, the Pre-Santonian sediments forming the Okigwe-Abakaliki anticlinorium and creating structural lows resulting in the Anambra and Afikpo basins. Thus, Anambra Basin was formed to the west and Afikpo to the east of the anticlinorium (Ogala, 2011; Agagu *et. al*., 1982).

The Anambra Basin is located south of the regionally extensive Northeast-Southwest trending Benue Trough. The basin has about 5km thickness of Upper Cretaceous Tertiary sediments characterized by enormous lithologic heterogeneity in both internal and vertical extension which were derived from a range of paleo-environmental settings (Akaegbobi, 2005). The lithostratigraphic units in this basin include the earliest Campanian sediments of the Nkporo and Enugu Shales. This is overlain by the dichronous Campanian to Maastrichtian Mamu Formation a coal measure, the Mamu Formation is overlain by the Maastrichtian Ajali Sandstone. The Nsukka Formation dichronous Maastrichtian-Danian of the lower Paleocene is underlain by the Ajali Sandstone, these ended the Upper Cretaceous sediments of the Anambra basin. The Tertiary sediments in the basin include the Paleocene Imo Shale, Eocene Ameki- Nanka Formation and Oligocene- Miocene Ogwashi-Asaba which terminal is lithostratigraphic sediment of the Anambra basin (figure 2).

The four outcrops locations used in this study, two from Nkporo Shale at Leru junction and Ivo River Enugu-Port Harcourt express and two were obtained from Enugu Shale, at Emene Junction and Enugu New Market, Enugu-Onitsha Highway (figures 3-6).

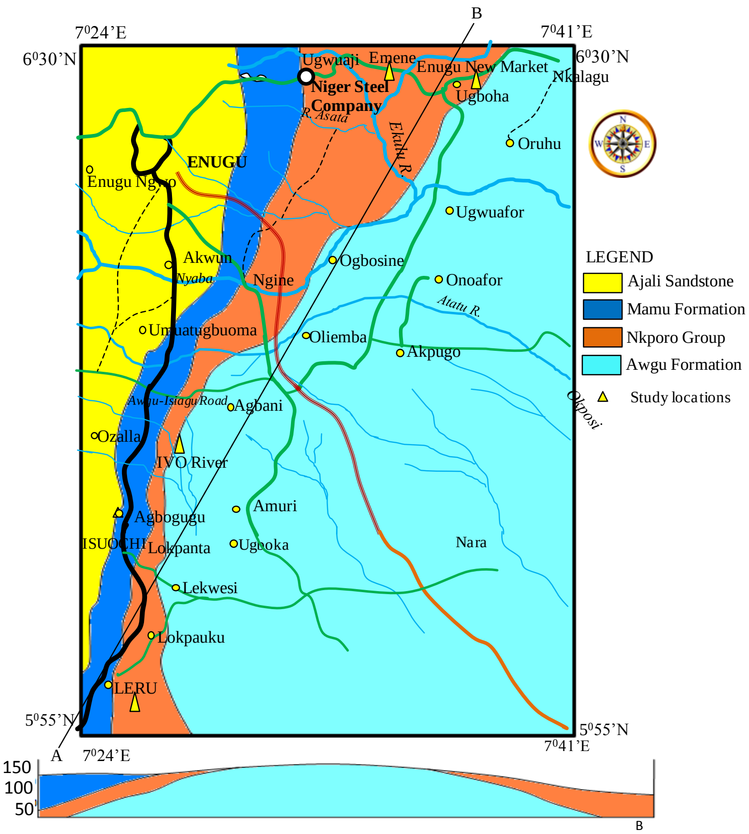


Figure 2: Geology Map of the Area (modify after NGSA, 1994).

3. **Materials and methods**

In this study, 4 samples were obtained from two Formations in the study area (Fig. 1 and 2) representing Nkporo shale deposit (Leru and Ivo River) and Enugu Shale deposit (Emene junction and Enugu New Market) source rocks in Anambra basin Southeastern Nigeria. Each sample was subjected to organic geochemistry to analyze for TOC using a LECO CS125 Carbon

Analyser and Rock-Eval parameters using Rock Eval LECO 6 machine module version. The following parameters; S_1, S_2, S_3, HI, OI and T_{max} values were obtained (Table 1). Parameter S_1 is the amount of free hydrocarbon (mg HC/g rock) liberated at 300ºC (without cracking the kerogen). S2 is the amount of hydrocarbon released from cracking of kerogen (mg HC/g rock) and heavy hydrocarbons during temperature programed pyrolysis (300– 600ºC) and represents the existing potential of a rock to generate petroleum. Peters et al (1994), Ogala (2011) and Nady et. al. (2015) believed that S_2 is a more realistic TOC because TOC includes dead carbon incapable of generating petroleum. S_3 represents the amount of CO_2 from breaking carboxyl groups and other oxygen-containing compounds in kerogen, obtained at 300– 390ºC. TOC was determined as follows; about 200 mg of the pre-cleaned shale was crushed and accurately weighed into clean LECO crucibles. The rocks were then de-mineralized by hot 10% HCl and afterwards washed repeatedly with distilled water. After drying at 60ºC the crucibles were automatically introduced into the furnace for combustion and measurement of the organic carbon content. The hydrogen index (HI) is the normalized S_2 value (S_2/TOC), expressed in mg HC/g TOC. The oxygen index (OI) is related to the amount of oxygen in the kerogen and is the normalized S_3 value (S_3/TOC), expressed in mgCO_2/g TOC. The production index (PI) shows the level of thermal maturation. The S_2/S_3 values indicate the type of organic matter for low to moderately mature samples (Nady *et al*., 2015).

Figure 3: Lithological unit at location one: Leru junction.

Figure 4: Lithological unit at location two; Ivo River along Agwu Isiagu road.

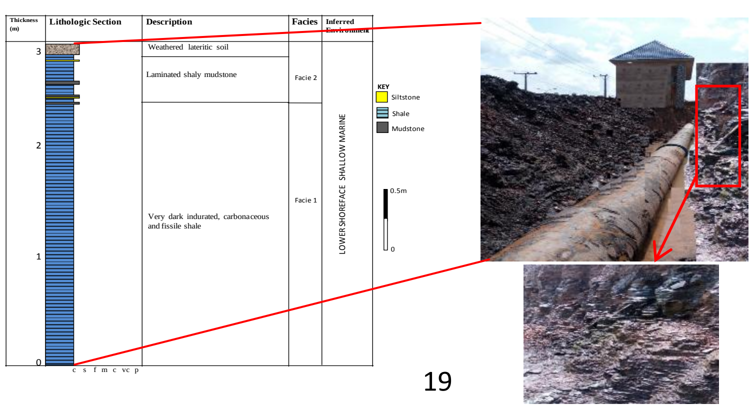


Figure 5: Lithological unit at Location three at Emene junction Enugu-Port Harcourt expressway.

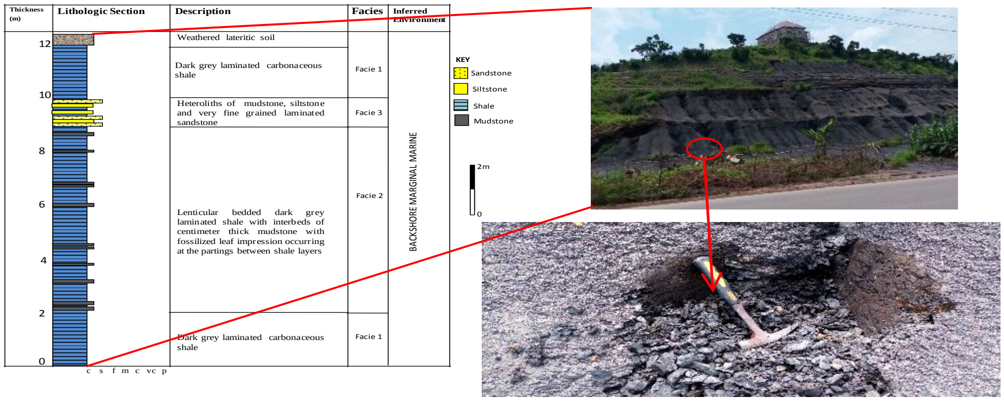


Figure 6: Lithological unit at Location four at Enugu New Market

4. Result and Discussion

Table 1: Rock-Eva Pyrolysis and TOC Results of samples collected from Nkporo and Enugu Shales.

Formation	Location	TOC	S_1	S_2	S_3	$T_{MAX K}$	HI	OI	S_2/S_3	$S_1/TOC*100$	PI	$GP=S_1+S_2$
Nkporo shale	Leru Junct. (11369)	2.05	0.03	0.95	0.28	438	46	14	3.4	1	0.03	0.98
	Ivo River (11368)	4.77	2.21	22.90	1.70	428	480	36	13.5	46	0.09	25.11
	Average values	3.41	1.12	11.925	0.99	433	263	25	8.45	23.5	0.06	13.045
Enugu shale	Emene Junct (11367)	2.41	0.14	3.86	0.39	435	160	16	9.9	6	0.04	4.00
	New Mkt (11366)	2.69	0.04	1.35	0.27	432	50	10	5.0	1	0.03	1.39
	Average values	2.55	0.09	2.605	0.33	433.5	105	13	7.45	3.5	0.035	2.695

4.0 Organic geochemical method

In the source rock evaluation, geochemistry is a valuable tool that finds out the determination of the organic carbon richness of the shale samples, type of organic matter and thermal maturity status of the organic matter present in the samples.

4.1 Total Organic Content (TOC)

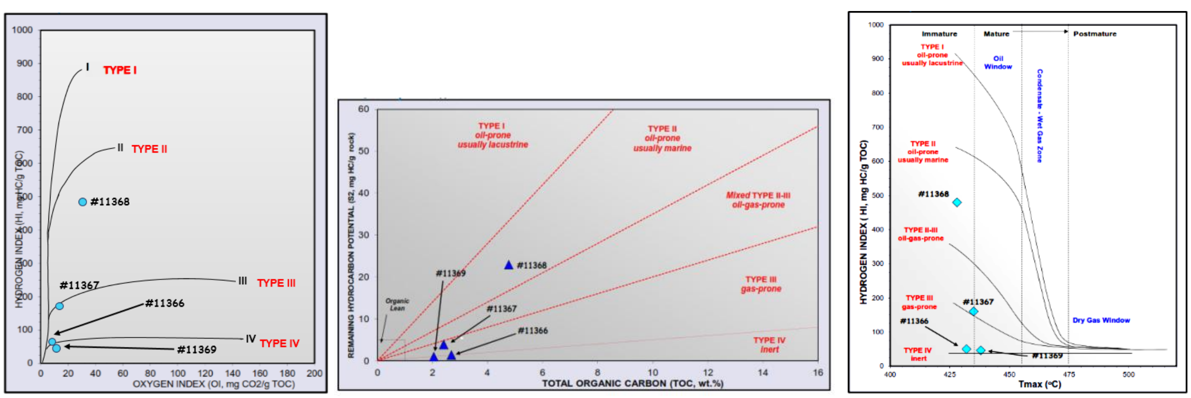
The aim of TOC is to determine the percentage by weight of the organic carbon content in source rock. That is the measurement of its potential to generate hydrocarbon. Based on the percentage organic content, the following source rock ratings were used as standards; Peter and Tisolt according to; Mohamed et al. (2014), Nady *et al.*, (2015), Uzoegbu *et al.*, (2014), Omoboriowo *et al.*, (2011), Iheanacho (2010), Passey *et al.*, and Ojo *et al*., (2009) .

The Total Organic Content (TOC) of the shale sample from Nkporo Shale (11369 and 11368) ranges from 2.05 to 4.77wt% with an average of 3.41wt%. And that of Enugu Shale (11367 and 11366) values 2.69 and 2.41wt% with an average value of 2.55wt%, these values exceeded the threshold value of 0.5%wt of 0.5wt%, the analyzed sample values indicate good-very good TOC for the Shales, using the standard threshold values

4.2.2 Organic Matter Type (Quality)

Van Krevelen coalification diagram was used to present the 4 principal types and evolution path ways of kerogen using *Hydrogen Index* (*HI*) and *Oxygen Index* (*OI*) from Rock-Eval pyrolysis analytical result.

The HI against OI plot was used to determine the kerogen type (Fig. 2), which shows that the analyzed Nkporo samples (11369 and 11368) were plotted along kerogen of Type IV and Type III path ways, while the analyzed Enugu samples (11367 and 11366) were plotted between Type II/III and Type III kerogen (figure 7a).



(a) (b) (c)

Figure 7: The Kerogen Type diagram of Nkporo and Enugu Shales **(a)**, Genetic kerogen quality of Nkporo and Enugu Shales **(b)** and Thermal maturity with kerogen quality of Nkporo and Enugu Shales **(c)**.

The organic matter type in a sedimentary rock, among other conditions, influences to a large extent the quality and quantity of hydrocarbon generated due to different organic matter type convertibility (Uzoegbue *et al*., 2014). The kerogen type was also deduced from the Hydrogen

Index (HI) values as follows; the Nkporo shale samples (11369 and 11368) ranges from 46 to 480mgHC/g TOC indicating Type IV and II respectively with the average value of 263mgHC/g TOC, whereas Enugu shale samples (11366 and 11367) ranges from 50 to 160mgHC/g TOC, with an average value of 105mgHC/g TOC indicating Type III and III (figure 7a). However, the average HI in a rock interval is best determined from the slope of a regression line on a graph of S_2 against TOC Langford and Blanc-Valleron standard rating (Olumuyiwa, 2015; Johannes, 2006; Nady *et al*., 2015). In this study, the plot of S_2 against TOC (figure 7b) were used to determine the kerogen Type and inferred the environment of deposition. This shows that the Nkporo shale samples (11369 and 11368) fall within Type IV (gas-inert) Terrestrial environment and Type II (oil prone) Marine environment. Whereas the Enugu shale samples (11367 and 11366) falls between transition line of Type II-III and Type III (oil-gas prone, predominantly gas with little oil) from mix Marine-Terrestrial environments and between Type III-IV (gas-inert) from Terrestrial environment.

4.2.3 Thermal maturation

The generation of petroleum from the organic matter during its burial history is a part of the overall process of thermal metamorphism of organic matter. The concentration and distribution of hydrocarbons contained in a particular source depend on both the type of the organic matter and its degree of thermal alteration (Nady *et al*., 2015). The thermal maturity level of a rock sample has been determined basically by the study of the geochemical parameters as Rock– Eval pyrolysis temperature, the maximum temperature (T_{max}), from standard values (Nady *et al*., 2015 and Olumuyiwa, 2015). This shows that from the result of Table 1, the samples from Nkporo shales (11368 and 11369) with T_{max} of 428 and 438°C were in immature and early mature respectively. Whereas the Enugu shale samples (11366 and 11367) with T_{max} values 432 and 435°C were in immature and early mature state respectively of the organic matter contained in the source rocks.

This is also confirmed with Salufu *et al*., (2015), Nady *et al*., (2015) and Olumuyiwa (2015) in the pyrolysis data kerogen classification diagram using the HI versus T_{max} plot for kerogen type and maturity level (figure 7c), which shows that the analyzed Nkporo Shale (11368 and 11369) samples were plotted in the immature zone of type II and early mature zone of III/IV. While the analyzed Enugu Shale (11366 and 11367) samples were plotted in the immature zone of Type III (gas prone) and marginally mature zone of Type II-III (oil-gas prone).

5. Discussion

From field observations, the Nkporo deposits were subcrops that are found below the surface, whereas Enugu shale deposits were outcrops from the surface. The inferred environment was shallow and marginal marine environment based on their lithology, coarsening upward sequence in both shales. The both shales were very fissile but Nkporo shale is more indurated in nature than Enugu shale which may likely be from the exposures.

From the Quantity of organic matter present, both Nkporo and Enugu shales samples were from a very good source rock, because they all exceeded the standard threshold of the 0.5wt% for fine-grained rocks capable of generating hydrocarbon.

Potential source rock: The both shales were found in suboxic environment which were also confirmed from their oxygen index values (very low).

From the Quality of organic matter present, Nkporo shale contain Type II and Type IV, this means that it is more prone to oil than little or no gas, which was also seen in the high content of its Hydrogen Index value. Whereas the Enugu shale consists of Type II-III and Type III which means that it is predominantly gas with little oil. The type of the organic matter present in these studied rocks determines the type of environment of their deposition. However, The HI/OI shows that the Nkporo shale samples (11369 and 11368) contain type IV and II kerogen. The general environment of deposition for type II kerogen is marine (11368 from an absolutely marine environment). Type II kerogen is composed of mainly plankton with some contributions of algae. These samples are an oil-prone source rocks. The type IV kerogen is from terrestrial environment, composed of dead carbon from land plants capable for dry gas or no petroleum. Meanwhile the Enugu shale samples (11367 and 11366) contain generally type II-III and III kerogen. The general environment for these types of mix kerogen are mix marine and terrestrial environment capable of generating abundant gas with little oil. Thus, both Nkporo and Enugu shales were deposited on marine and terrestrial environment.

From the Thermal maturity, Nkporo samples range from immature to generate mainly oil and early mature to generate inert gas. Whereas Enugu samples also range from immature to generate mainly gas and early mature to generate more gas with little oil. The immature and early mature ability of these shales might be from the low rate of the basin subsidence at the time of deposition of the Formation according to Salufu et al (2015). However Onyekuru S.O. uses the plot of HI against OI in Van Krevaline diagram to depict the maturity of shales samples by plotting within the origin of the graph; in this study part of Nkporo and Enugu shale (11369 and 11366 respectively) were plotted within the origin indicating that they are highly mature.

From the Hydrocarbon potential, S_2 corresponds to hydrocarbons released as a consequence of the thermal cracking of kerogen. It represents the amount of petroleum that might be generated from the source rock if thermal maturation continues. Thus, it is a key measurement in terms of petroleum potential. In this study, S_2 values of 0.95 and 22.90 petroleum potential (Leru and Ivo River) from Nkporo Shales indicate that they have poor and excellent capabilities to generate petroleum if thermal maturation continues. And 1.35 and 3.86 values of petroleum potential (New market and Emene) for Enugu Shales also indicate poor and fair capabilities to generate petroleum if thermal maturation continues. Also from the Generating potentials, both Nkporo and Enugu shales have capacity to generate oil and gas, but Nkporo has excellent potential to abundant oil than gas, whereas Enugu has fair potential to predominantly gas than oil.

6 Conclusions

In conclusion, four (4) shale samples from Nkporo and Enugu were analysed, the quantity of organic matter and high organic richness for a potential source rock.

The Quality of organic matter; the Nkporo shale samples (11368 and 11369) contain type II and type IV kerogen; The Enugu shale samples (11367 and 11366) contain type II-III and type III kerogen both from marine and terrestrial environment.

The Thermal maturity shows that Nkporo shale (11368 and 11369) shows immature and early mature to generate oil and inert gas respectively. Enugu shale samples (11367 and 11366) show early mature and immature to generate oil-gas, and gas prone respectively. However, in Van Krevaline diagram (figure 7a), part of Nkporo and Enugu shale (11366 and 11369) were plotted within the origin indicating that they are highly mature.

The similarities in their geochemical results such as TOC, Tmax, HI, maturation, kerogen type show that the both Shales come from the same source rock.

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