Evaluation of the Shale Gas Potentials of Ekenkpon Shale, Calabar Flank, Southeastern Nigeria

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ABSTRACT

The Cenomanian-Turonian Ekenkpon Shale of the Calabar Flank were analysed to assess their potentials for shale gas. To achieve this, core samples were collected from shallow exploratory wells and complemented with samples from surface outcrops. These were subjected to sedimentological (lithofacies analysis, thin section studies) and geochemical analyses (total organic carbon, rock-eval pyrolysis, x-ray fluorescence and fluid inclusions stratigraphy). The Shale is light to dark grey, moderately hard, laminated (and highly fissile in outcrop exposure), silty in places, calcareous and fossiliferous. There is lateral and stratigraphic variation in the Ekenkpon Shale ranging from shale, mudstone, carbonate and siltstone. The mineralogy is dominated by clay minerals (68%), silica (25.75%) and carbonates (11.66%) with high presence of pyrite and heavy minerals. The average total organic carbon (TOC) is 1.43wt%. The maximum temperature (Tmax) of hydrocarbons expulsion upon thermal cracking range from 429°C to 457°C (av. 440 °C). The sample showed early to peak maturity having vitrinite reflectance values of 0.56% to 1.06% (average 0.77%) and therefore falls within the oil and gas window. From the above results Ekenkpon Shale has moderate organic richness, sufficient thermal maturity and predominantly type III kerogens. Fluid inclusion species of benzene tends to be increasing with depth suggesting proximity to pay zone. The sedimentological characteristics of the Ekenkpon shale (micro scale fabric anisotropy and brittleness) and its geochemical/mineralogical properties are reminiscent of some of the United States commercial gas shales, having favourable conditions and reservoir characteristics to qualify it as a potential shale gas play. The increase in maturity and FIS species with depth suggests that deeper sections of the Ekenkpon Shale presents greatest potentials for commercial accumulation of shale gas plays. Therefore, future exploration strategies should focus deeper sections since the gas potential indices got better with increasing depth across the formation.

Keywords: Calabar Flank, Ekenkpon Shale, Fluid Inclusions, Geochemistry, Lithofacies, Sedimentology, Shale gas, Stratigraphy.

INTRODUCTION

Adjacent to the petroliferous Niger Delta basin, the Calabar Flank is located along the corridor of the South Atlantic Marginal basins. Since the Separation of Africa and South American plates vis-a-viz the development of the aulacogen (The Benue Trough), the basin evolution has witnessed several tectonic activities. These activities influenced the sedimentary facies distribution and in concert with sea level oscillations, continental and marine influences have been recorded within the Calabar Flank and Nearby Anambra basin and Benue Trough. The oldest sediment in the Calabar Flank is the continental Awi Formation believed to have been deposited unconformable following the creation of the basin during Early Cretaceous (Neocomian – Aptian). This was followed by marine incursion into southern Nigeria through the windows of the Calabar Flank with the deposition of shallow marine platform carbonate (Mfamosing Formation). Since then, deepening of the basin allowed for accumulation of shales from the Cenomanian to Maastrichtian times. The records of global Cretaceous paleoanoxia seem to have been recorded in the Ekenkpon Shale (Petters 1978; Nyong and Ramanathan, 1985) and this makes this formation a suitable candidate for the investigation for shale gas potential. Often generated and trapped within a very low permeability organic rich shale, shale gas deposit differs from convention shales that are primarily regarded as source and seal rocks. This has focused attention on shale gas reservoirs as 'unconventional' systems (Hunt 1996,

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Jarvie *et. al.,* 2007, Tewari *et. al.*, 2016). In the past two decades, efforts have been focussed on assessing the potential of inland basins in Nigeria to buffer the hydrocarbon (crude oil) reserves of the nation, however, since the emergence of shale gas exploration and its exploitation, more effort and attention have shifted towards its development. To date, no study on the evaluation of shale gas potential has been carried out in the Calabar Flank and indeed southern Nigeria. This is because shale gas development in Nigeria is in its earliest stages as yet little or no targeted publication and reserved estimates on Nigerian shale gas resources are documented. In today's world, shale gas is swiftly replacing conventional fuel to suffice the ever-growing energy requirements. The fluctuating oil price, high input cost in exploration and production of hydrocarbons and the relative abundance of natural gas resources have changed the demeanour towards unconventional shale gas exploration. The numerous applications of natural gas and its green environmental conditions imply that shale gas will play an important role in fitting into current fossil fuel policies and in meeting the near future energy demands. This assessment takes into account current changes such as the drop of oil prices (having an impact on the gas market), the variations in energy policy and climate policy of regions and countries. In other climes, shale gas has been integrated into national energy mix accounting for about 8% of United States of America's total natural gas production (Warlick, 2006). The success of the Barnett Shale play in Texas has activated research for shale gas resources across the United States, Canada, Europe, Asia and Australia. Nigeria has the immense potential of unconventional natural gas resources considering the enormous shale deposit across the sedimentary basins in the country i.e., coal bed methane (CBM), tight gas and shale oil and gas (Obaje *et. al.,* 2004; Essien *et. al.,* 2005; Abubakar et. al., 2008, Ekpo *et. al.,* 2013; Boboye and Okon, 2014). There is limited understanding of the evolution of the subsurface rocks in the Calabar Flank due largely to inadequate subsurface data, as most research were previously carried out on exposed outcrop sections. Although, few reports are available on the organic richness and thermal maturity of the sediments, detailed organic geochemical investigations on the organic matter quantity and quality, origin of the organic matter as well as the burial and thermal histories of source rocks and the timing of hydrocarbon generation (Essien *et. al.,* 2005, Odumodu, 2012, Ekpo *et. al.,* 2012, Boboye and Okon, 2014), within the framework of evaluation of the shale gas resource, these accounts are grossly inadequate. Commercial exploration of these resources can effectively make the natural gas curve more elastic. The identification of shale resource plays in Nigeria would present a source of carbon-based energy with the lowest carbon dioxide emissions (Johnson *et. al.,* 2014). The general objective of this contribution is to assess and analyse the potential of shale gas in Nigeria, particularly the Ekenkpon Shale, Southern Nigeria. The analysis focusses on the geochemical characteristics and related factors that are responsible for economic accumulation of shale gas resource. Mention is made in this study about associated difficulties and challenges involved in the development and exploitation of shale gas resources with examples from well-known deposits across the globe.

GEOLOGICAL SETTING AND STRATIGRAPHY OF THE CALABAR FLANK

The term Calabar Flank was first introduced by Murat (1972) when he attempted describing sediments southeast of the Benue Trough. To the south, it is delimited by the north-eastern extent of the Tertiary Niger delta at the Calabar Hinge line and to its northern boundary by the Oban Massif. It is also separated from the Ikpe platform to the west and in the east, it extends up to the Cameroun volcanic line (Figure 1). 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 (Reijers and Petters, 1987). Its origin is strategically related to the break-up of Gondwanaland supercontinent and the opening of the South Atlantic and the associated Indian Ocean (Benkhelil and Guiraud, 1980). The failed rift (aulacogen), referred to as the Benue Trough is linked to the West and Central African Rift System (WCARS) and developed into a mega-stratigraphic basin within which the Calabar flank is viewed as a sub-basin (Whiteman, 1980; Genik, 1993; Nwajide 2013).

Structurally, the Calabar Flank consists of basement horsts and grabens that are aligned in a NW – SE direction

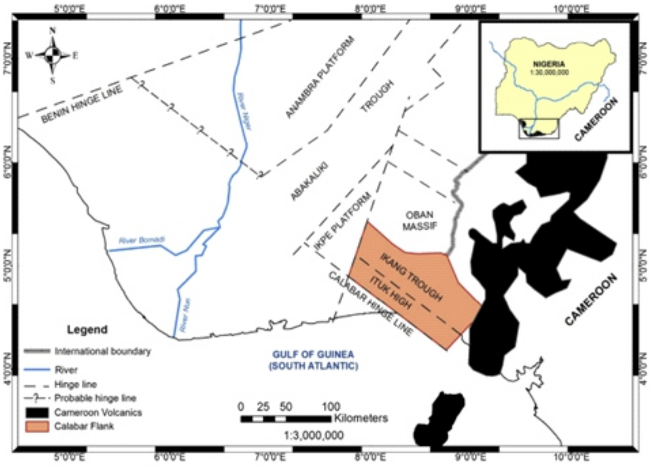


Figure 1: The map of Niger Delta showing structural elements and the Calabar Flank in relation to other adjacent sedimentary basins in Southeastern Nigeria (Adapted from Nyong and Ramanathan, 1985).

like other South Atlantic marginal basins in West Africa (Reijers and Petters, 1987) and orthogonal to the regional trend of the Benue Trough. The Calabar Flank shows striking stratigraphic similarities with other marginal basins of the South Atlantic. Northwest-southeastern trending basement structures underlie the Calabar Flank and define the ltu High and the lkang Trough (Figure 2), thus relating the Calabar Flank to other South Atlantic Cretaceous marginal basins (Angola and Gabon) with similar horst-and-graben structures (Ekpo *et. al.,* 2012). The sedimentary succession on the Calabar Flank is mostly of Cretaceous age, comprising ancient fluvio-deltaic sandstones, the Awi Formation, which is predominantly Sandstone with intercalations of shales/mudstone, dated to be of Neocomian-Aptian age.

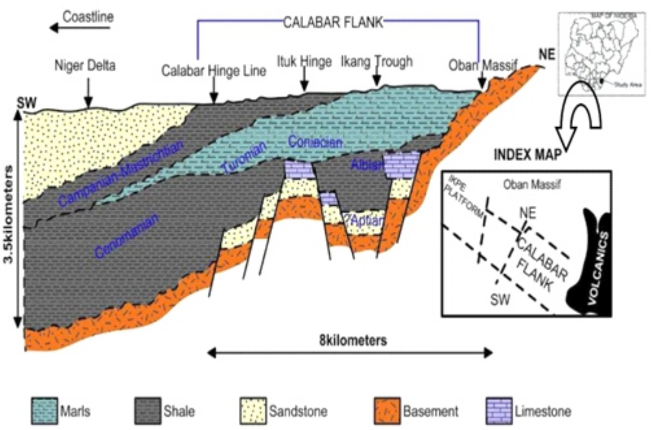


Figure 2: Structural elements and conceptual subsurface distribution of Cretaceous sediments in the Calabar Flank (After Nyong and Ramanathan, 1985).

This stratigraphic unit unconformably overlies the basement complex; and is overlain by the Odukpani Group. The Odukpani Group comprises; the Mfamosing Limestone, the Ekenkpon Shale which is our main area of focus in this research study and the New Netim Marl, which are all exposed near the geologically famous Odukpani junction. The Awi Formation is directly overlain by platform carbonates of the Mfamosing Limestone Formation (Petters, 1982), deposited during mid-Albian times. The Mfamosing Limestone is overlain by the thick sequence of black to grey shale unit, the Ekenkpon Formation (Reijers and Petters, 1987). The shale is characterized by minor intercalation of marly limestone, calcareous mudstone and oyster beds. It was deposited during the late Cenomanian-Turonian times (Reijers and Petters, 1987). The Ekenkpon Shale is overlain by a thick marl/shale unit: The New Netim Marl (Petters *et. al.,* 1995). This unit is nodular and shaly at the base and is interbedded with thin layers of shale in the upper section (Petters *et. al.,* 1995). Foraminifera results suggest early Coniacian age for this marl unit (Nyong and Ramanathan, 1985). The New Netim Marl is unconformably overlain by carbonaceous dark grey shale, the Nkporo Formation (Reyment, 1965). The shale unit was deposited during the late Campanian-Maastrichtian times after the widespread Santonian deformational episode in a wide range of environmental settings including shallow open marine, paralic and continental regimes. The Nkporo Shale caps the Cretaceous sequence in the Calabar Flank (Figure 3). The Nkporo Shale sequence is overlain by a pebbly sandstone unit of the Tertiary Benin Formation. Some stratigraphic divisions have included the Benin Formation as part of the Calabar Flank (Petters *et. al.,* 2010), while others are of the view that the basin forming processes of the Calabar Flank ended in the Cretaceous (Okon, *et. al,* 2017). The Benin Formation is characterized by yellowish-brown and white continental sand, alternating with pebbly layers and few clay beds (Reyment, 1965). The total sediment thickness in the surfaces of the Calabar Flank is over 3500m (Ekpo *et. al.,* 2012).

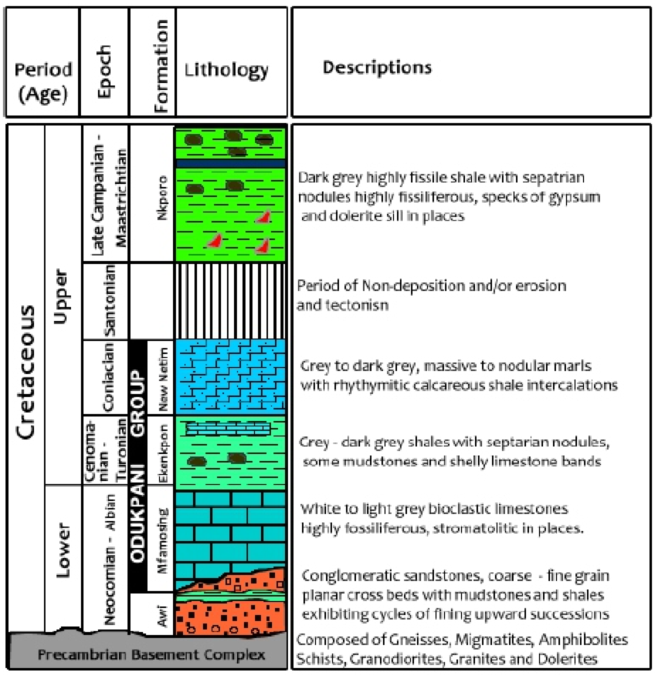


Figure 3: Stratigraphic chart of the Calabar Flank (Okon et. al., 2017).

The results from Ituk-2 and Anua-1 wells drilled in the Calabar Flank shows that the thickness of the Ekenkpon Shale ranges between 580-817m (Odumodu, 2012) in these two wells. The geological location of the Calabar Flank with respect to other southern Nigerian sedimentary basins is illustrated in the geological map of Nigeria in Figure 4.

MATERIALS AND METHODS

The data set for this research consists of cored shale samples from shallow wells and outcrop samples within the Ekenkpon Shale of the Calabar Flank. Samples were collected from eleven shallow exploratory wells that drilling activities penetrated the Ekenkpon Shale and four surface samples from the outcrops. Field activities traverses several communities including Odukpani, Mfamosing, Abiati and Etankpini, Oberekkai and New Netim covering a considerable portion of the Ekenkpon Shale, all within the Calabar Flank (Figure 4). The methodological approach used to accomplish this research is summarized in the flow chart (Figure 5). It includes desk study after which intensive field study was carried out. Core sampling and lithologic descriptions was followed by laboratory (geochemical) analyses. After field mapping, the samples were air dried and archived. This was followed by sampling (at 5 m interval) and lithologic description of core sampled collected from eleven (11) boreholes. Representative samples were cut into thin section for optical analysis of the mineral constituents. The samples were further crushed into fine powder (60-100 mesh) and analysed for total organic carbon (TOC) content using titration method. Approximately 0.5 grams of the samples were weighed, treated with concentrated hydrochloric acid to remove carbonates. Using the results for TOC analysis, potential source rock samples (19 Nos) were selected for further analysis using a Rock-Eval 6 pyrolyser.

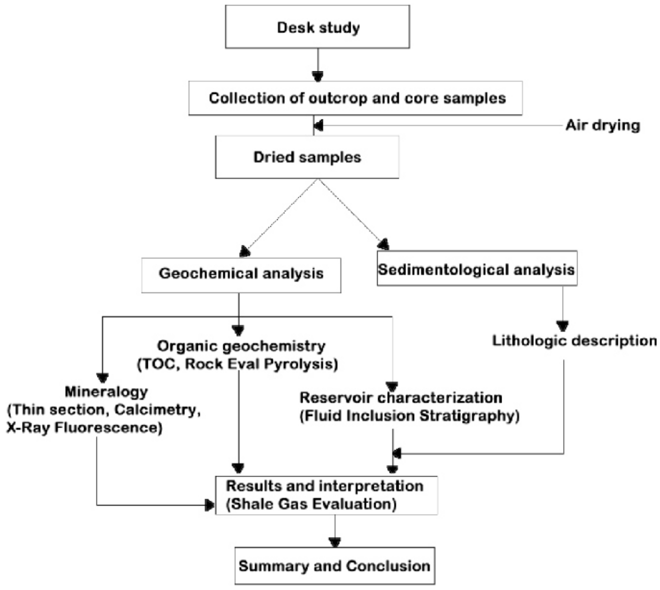


Figure 5: Simple flow chart describing the procedural approach for this research.

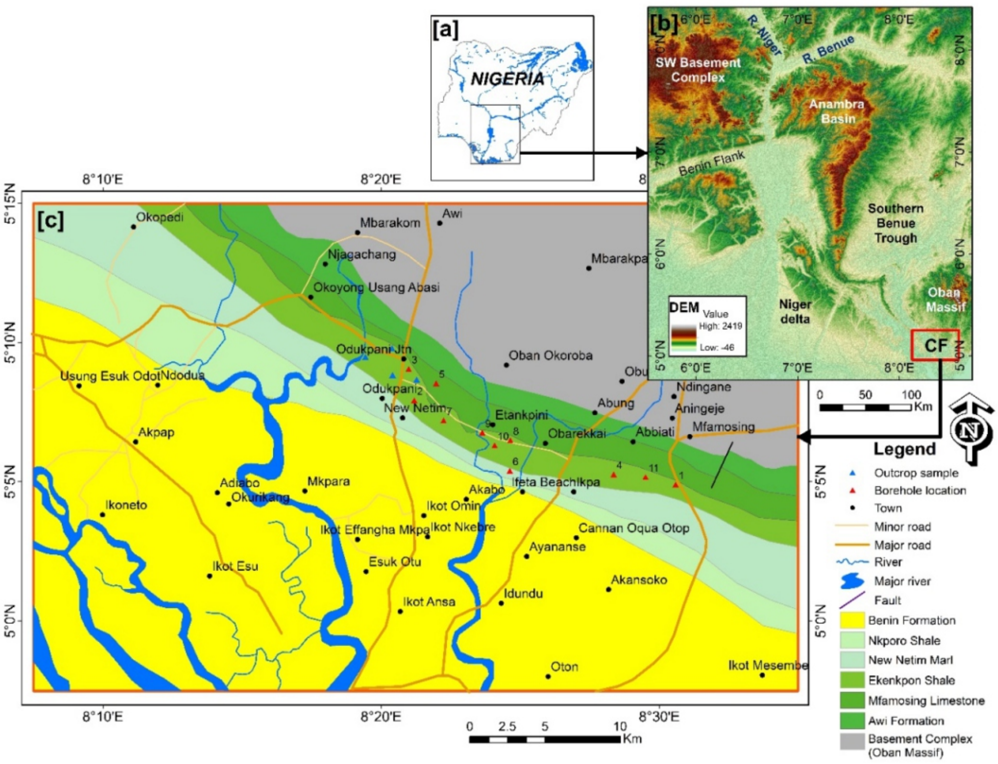


Figure 4: Geological map of the study area showing sample locations (a – Map of Nigeria showing southern Nigeria, b – Digital Elevation Model of southern Nigeria showing the study area, c – Calabar Flank).

Programmed pyrolysis was calibrated from 300 °C to 650 °C at a heating rate of 25 °C/min under nitrogen, and residual carbon was oxidized in an oxidation oven. This phase began with an isothermal stage at 300 °C and the temperature was then increased at a rate of 25 °C /min to 750 °C. The amount of hydrocarbon released during pyrolysis was determined using a flame ionization detector (FID), while CO and CO_2 released during the process were continuously measured with an online infra-red detector. This methodology is predicated upon the fact that hydrocarbon generation and maturation processes are highly controlled by time, temperature, pressure, depth of burial etc. Therefore, the experimental temperatures as described above, were set moderately higher than normal subsurface conditions, so that appreciable reactions for the generation of hydrocarbons could occur in a practically short time and the quantity of generated hydrocarbons relative to the total potential of the source rock can be estimated (Peters, 1986; Espitalie, 1986; Jarvie *et. al.,* 2007). The samples were also subjected to calcimetry analysis and x-ray fluorescence analysis. Finally, the samples were subjected to fluid inclusion stratigraphy (FIS) analysis involving rapid complete analysis of volatiles trapped as fluid inclusions in rock samples using quadrupole mass analysers attached to an automated high vacuum sample introduction system (Hall, 2008). The technique documents the presence and relative bulk abundance of ionized volatile fragments with mass to charge ratio of $1 \leq m/z \leq 180$ that have been released from fluid inclusions by crushing of natural samples. This includes most geologically important inorganic species as well as organic species with less than or equal to 13 carbon atoms. The resulting analysis of the petroleum fraction is comparable to the low molecular weight fraction of a whole-oil gas chromatographic- mass spectrometric (GCMS) analysis (without devolatilization of the gas fraction); hence the major classes of hydrocarbons (e.g., aromatics, naphthene and paraffins) are represented. Unlike GCMS where the quadrupole is front-ended with a GC, boiling point separation is not achieved and all species are analysed simultaneously. The FIS analysis was carried out at the Schlumberger laboratory in France.

RESULT AND INTERPRETATIONS

Lithologic Description

Following outcrop study, lithological description of the Ekenkpon Shale was carried out. This was integrated with the core description to develop a stratigraphic framework for the Ekenkpon Shale (Figure 6 a-f). Generally, the shale beds exhibit exhibits a light to dark grey colouration, laminated to blocky parting lineation, fissile in most weathered exposed sections. The shale may become silty in places and clayey in other places. The rocks are tight, compacted but with micro fractures visible. The laminations in the shales are due to presence of thin layers of silt and clay, and sometimes colour alteration from light grey to dark grey due to presence of organic matter in differential quantity. The presence of laminated to blocky shale units reflects both uniform and differential rate of sedimentation. The lower part of the formation is composed of thin layers of fine to medium grained limestone and sandstone intercalations (Figure 6E). Thick sequence of carbonaceous and non-calcareous shale is the main composition of this formation. Typically, from the base to the top the Ekenkpon Shale consist of light grey - dark grey shale intercalated with thin limestone band. The shale is highly fossiliferous with abundant pelecypod, ammonites, oysters and trace fossils in certain horizons. It is capped by weathered lateritic claystone towards the top of the unit. Stratigraphically, the Mfamosing Limestone underlies the Ekenkpon Shale as indicated in most well sections (Figure 7).

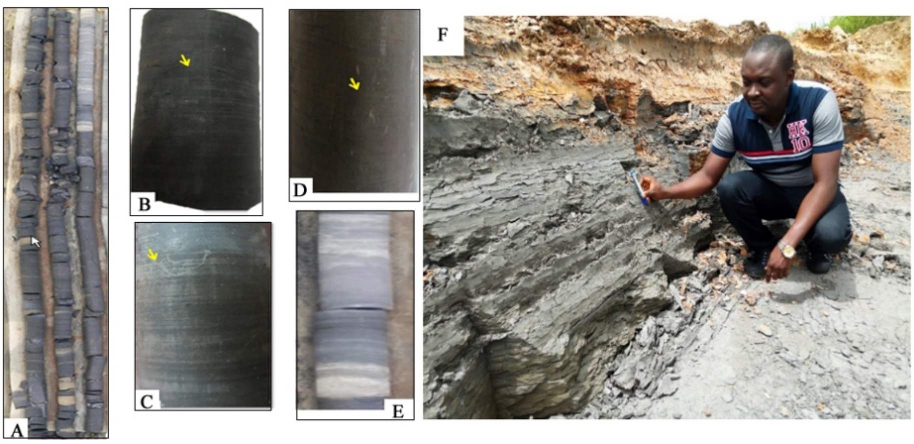


Figure 6 a-f: Core samples of Ekenkpon Shales from Well EKS10. **A**. cores of Ekenkpon Shale Formation; **B.** Laminated dark grey shale, **C.** Laminated light grey shale, colour differentiation due to mineralogy or presence of organic matter; **D.** Dark grey shale; **E.** Limestone/sandstone-shale Intercalation. **F.** Photograph showing shale laminations and natural fractures (Location: L1).

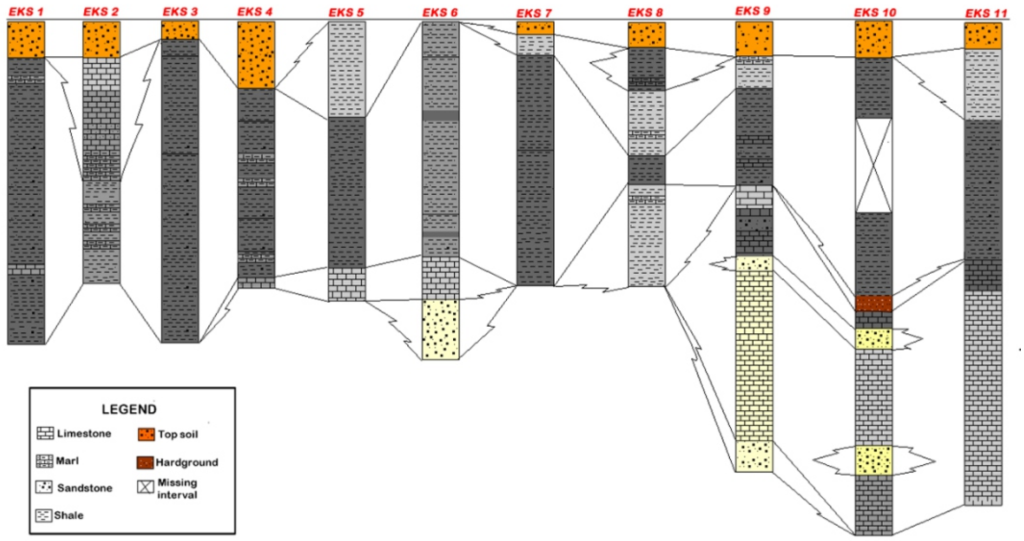


Figure 7: Wells Correlation showing the continuity of Ekenkpon Shale, Calabar Flank, South-eastern Nigeria

Organic Geochemistry

This section presents the organic matter richness, petroleum generation potential, type of organic matter and maturity evaluation of the Ekenkpon Shales. A total of 112 samples from different depth interval of the Ekenkpon Shales were analysed for TOC. A total of 19 samples were selected for Rock Eval Pyrolysis. All the samples were analysed using Rock Eval Pyrolysis-6 to characterize the geochemical properties of Ekenkpon shales. The shale unit varied in colour and organic matter, with the dark grey shale found to be rich in TOC The organic richness, kerogen type, thermal maturity of Ekenkpon Shale is investigated. Fluid inclusion stratigraphy (FIS) was integrated with the results of conventional organic geochemistry of the Ekenkpon Shale, for the first time, and used to evaluate the hydrocarbon content of the unconventional reservoir rock.

Total Organic Content (TOC)

The summary of the results of TOC analysis and the original TOC (TOCO) of samples calculated from measured TOC values, using numerical methods (Jarvie and Lundell, 1991; Jarvie, 2004) is presented in Table 1. The result shows that TOC values range from 0.11 wt. % to 4.15 wt.% with an average of 1.94 wt.%. The original TOC differs from the present-day TOC (TOCP) content (TOCP/0.64 =TOCO) and presents highly values for the organic matter content. The calculated original TOC (TOCO) values range from 0.04 to 6.48 wt.% (av. 2.12 wt.%) for the analysed Ekenkpon Shales samples. Demaison and Moore (1980) have noted that low energy, oxygen-depleted (anoxia) environments are best suited conditions for preserving organic matter. However, variations exist in the TOC concentration due to climatic variations during sediment deposition, organic matter decomposition and weathering effect. Also, post depositional diagenesis processes, increasing pressure and temperature with burial depth causes thermal alteration of organic matter (i.e. TOC loss with depth is due to maturation, hydrocarbon generation and expulsion) and contributes to TOC variations in source rocks (Jarvie *et. al.,* 2007, Wang and Carr, 2013, Jiang *et. al.,* 2016). Therefore, based on the criterion of minimum TOC abundance (TOC > 0.5%; Peters, 1986) for hydrocarbon source rock abundance evaluation, majority of the samples from Ekenkpon Shale maybe regarded as potential source rocks for the production of shale gas.

Table 1: Total Organic Carbon (TOC) and Total Organic Carbon-Original (TOCO of Wells in Ekenkpon Shale (wt.%).

J Ĝl I NAMES	ĜHĬ Ĉ	ĜHĬ Č	EKS3	EKS4	EKS5	ĜHĬ Ď	ĜHĬ Đ	EKS8	EKS9	EKS10	EKS11
Ì IN TOC (Wt. %)	ĆBĎD	ĆBDĊ	ĆBČČ	ĆBDČ	ĆBČĐ	ĆBĈĈ	ĈBČĊ	ĆBDÇ	ĆBDÇ	ĆBĈĐ	ĆBĈĈ
Ì ĖĴ TOC (Wt. %)	ČBDĈ	ĈBEE	ĈBČD	ÇBĈD	ČBĈĊ	ČBĎE	ČBDĐ	ČBČČ	ČBÇĐ	ĊBDĎ	ĈBEĎ
ĖIJĠ TOC (Wt. %)	ĈBÇČ	ĈBDĎ	ĆBĎĎ	ĈBEÇ	ĈBČĐ	ĈBDĊ	ĈBDE	ĈBDĈ	ĈBĊĊ	ĆBEĊ	ĆBDĊ
Ĭ ÞÖ Ö MǾŘ ÖŅİ Î FÎ QMÕÞ ÑŒ											
Ì Hİ TOCO (Wt. %)	ĈBĆĈ	ĆBDČ	ĆBĊÇ	ĆBDĈ	ĆBÇČ	ĆBĐĐ	ĈBDĐ	ĈBĊĈ	ĈBĆĎ	ĆBČĎ	ĆBĈĐ
Ì ĖĴ TOCO (Wt. %)	ĊBEČ	ĊBĈĆ	ĈBED	ĎBÇĐ	ĊBČČ	ÇBČĆ	ÇBĆĈ	ĊBĆE	ĊBĐĐ	DBDĎ	ĊBĆĎ
ĖIJĠ TOCO (Wt. %)	ČBČČ	ČBÇĊ	ĈBĆĊ	ĊBĆČ	ĈBEE	ČBÇĎ	ČBDĆ	ČBĈD	ČBĆE	ĈBĊE	ĈBĊĊ

Rock Eval Pyrolysis Analysis

The results of Rock eval pyrolysis is presented in Table 2. From the results, several Rock Eval parameters were calculated (i.e. hydrogen index (HI) defined as 100 × S_2/TOC; oxygen index (OI) defined as 100 × S_3/TOC; where S_3 is the CO_2 released during the pyrolysis). Both the measured and calculated parameters from Rock Eval pyrolysis, helps in determination of kerogen type, hydrocarbon generation efficiency and maturation. The Rock Eval Pyrolysis generated parameters are S_1, S_2, S_3 and Tmax whereas calculated parameters are hydrogen index (HI), oxygen index (OI), production index (PI), genetic potential (PI), vitrinite reflectance (VRo) etc. S_2 values from 0.01 to 2.38 mg HC/g rock. The samples show low to moderate oxygen index (OI) values ranging from 1 to 418 mg CO_2/g TOC. Hydrogen index (HI) values range from 0.0 to 306 mg HC/g TOC, and Tmax from 429 to 457 °C.

Genetic Potential (GP)

A genetic potential is the summation of the amount of free hydrocarbon (S_1) and the quantity of remaining thermally generated hydrocarbons (S_2). This can be mathematically expressed as (S_1+S_2) measured in mg/g of rock (Tissot and Welte, 1984). Genetic potential values and other source rock evaluations parameters are shown in Table 2. A fair to good genetic potential was observed in most of the studied samples. The average GP of the studied samples is 0.78 mg HC/g Rock, varying from 0 to 6.62 mg HC/g Rock. The maximum genetic potential of 6.62 mg HC/g Rock was recorded at the depth of 40m in well EKS10. A genetic potential of less than 2 mg HC/g Rock has little or no oil, but has potential for gas (Dymann et. al., 1996). The cross plot between GP and TOC shows that the samples have fair to good hydrocarbon generation potential (Figure 8). There is a linear correlation between the TOC and GP. This indicates that genetic potential of the formation increases with a higher TOC values.

Productivity Index (PI)

In assessing the potentiality of the source rock for shale gas, the quantification index of the proportionality between the hydrocarbons that already generated (S_1) from kerogen and the quantity of whole hydrocarbons that can be obtained from Kerogen, the productivity Index (PI) can be derived from Rock eval parameters using the relationship S_1/ (S_1+S_2).

The PI of the studied samples range from 0.04 to 0.34mg HC/g TOC and indicates in situ petroleum generation (Peters, 1986; Peters and Moldowan, 1993). The highest PI of 0.34mg HC/g TOC was recorded in Well EKS9 at the depth of 70m. The weathering processes or oxidation removes hydrogen and adds oxygen to the kerogen which can slightly alter the original PI value. Generally, the commercial gas shale producing horizons show PI values ranging from 0.6 to 1.5, however shales with greater than 0.1 PI can generate good quantity of hydrocarbon (Peters, 1986).

Hydrogen Index (HI) and Oxygen Index (OI)

This is an invaluable tool for determining the kerogen type. These indices are mathematically expressed as [(100 x S_2)/TOC] and [(100 x S_3)/TOC] for the hydrogen index and the oxygen index, respectively (Hunt 1996; Peters and Cassa, 1994). At early thermal maturity stages according to Tissot and Welte (1984), HI values of > 600 mg HC/g TOC and OI values <50 mg CO_2/g TOC; HI values 300–600 mg HC/g TOC and OI values <50 mg CO_2/g TOC; HI 200–300 mg HC/g TOC; HI values 50–200 mg HC/g TOC and OI values of 5–100 mg CO_2/g TOC; HI values of <50 mg HC/g TOC points to Type I kerogen, Type II kerogen, mixed Type II and III kerogen, Type III and Type IV kerogen respectively and will be used to evaluate the sediments of Ekenkpon Shale.

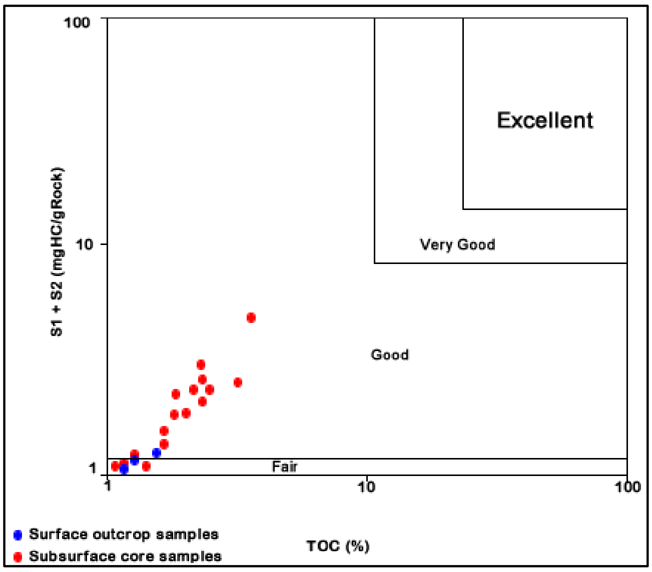


Figure 8: Cross plots of Generation Potential (GP) versus TOC (after Ghori, 2002).

With the HI values of Ekenkpon Shale ranging from 0 to 306 mg HC/g TOC (av. 79.10 mg HC/ g Rock). Boyer et. al., (2006) have noted that low HI values, say <125.34 mg HC/g TOC, indicates a greater potential to generate gaseous hydrocarbon. Clearly, from the cross plot between HI and OI of analysed samples (Figure 9) the predominance of type III, suggest gas prone kerogen (Hunt, 1996, Okon, 2011, Boboye and Okon 2014). It is important to note that, there is some progression with increasing maturity and type II kerogen with depth, as indicated by the single plot HI of 306 mg HC/g TOC and OI of CO_2/g TOC for sampled from Well EKS9 (70m depth). This position was also envisaged when Espitalie (1986) noted the increase of PI with burial depth.

Source rock Thermal maturity

Thermal maturity of a source rock is a critical component in its evaluation because of the relationship that exist

Table 2: Rock Eval Pyrolysis Results for Selected Samples of Ekenkpon Shale

WELL NAME	Depth (m)	TOC (%)	S_1 (mg/g)	S_2 (mg/g)	S_3 (mg/g)	ÖŃ (°C)	HI	OI	PI	S_3CO (mg/g)	PC (wt %)	RC (wt %)	MINC (wt %)	OICO	GP	VRo	S1/TOC	PC/RC (wt %)
L1	3	0.32	0	0.06	0.03	441	19	9	0.07	0	0.01	0.31	7.49	0	0.06	0.78	0.00	0.03
L3	2	0.03	0	0	0.03	440	0	100	0.14	0.01	0	0.03	7.19	33	0	0.76	0.00	0.00
L4	3	0.44	0.01	0.23	0.17	429	52	39	0.05	0.03	0.03	0.41	2.24	7	0.24	0.56	0.02	0.07
EKS4	20	1.05	0.06	0.59	0.54	440	56	51	0.09	0.26	0.1	0.95	2.17	25	0.65	0.76	0.06	0.11
EKS4	45	0.38	0.02	0.23	0.11	439	61	29	0.1	0.02	0.03	0.35	3.02	5	0.25	0.74	0.05	0.09
EKS5	40	0.21	0.02	0.15	0.21	438	71	100	0.11	0.13	0.03	0.18	7.48	62	0.17	0.72	0.10	0.17
EKS6	35	0.4	0.01	0.14	0.06	437	35	15	0.08	0.16	0.03	0.37	2.07	40	0.15	0.71	0.03	0.08
EKS6	50	0.05	0	0.01	0.02	438	20	40	0.04	0.02	0	0.05	6.75	40	0.01	0.72	0.00	0.00
EKS8	15	0.79	0.05	0.41	0.07	438	52	9	0.1	0.03	0.05	0.74	0.51	4	0.46	0.72	0.06	0.07
EKS8	35	1.13	0.07	0.85	0.06	443	75	5	0.07	0.03	0.09	1.04	0.75	3	0.92	0.81	0.06	0.09
EKS9	15	0.62	0.04	0.4	0.07	441	65	11	0.09	0.02	0.04	0.58	1.12	3	0.44	0.78	0.06	0.07
EKS9	45	2.06	0.56	2.38	0.34	451	116	17	0.19	0.05	0.26	1.8	2.65	2	2.94	0.96	0.27	0.14
EKS9	70	0.17	0.28	0.52	0.07	436	306	41	0.35	0.01	0.07	0.1	8.06	6	0.8	0.69	1.65	0.70
EKS10	10	0.9	0.07	0.43	0.06	441	48	7	0.14	0.03	0.05	0.85	0.14	3	0.5	0.78	0.08	0.06
EKS10	35	0.53	0.04	0.25	0.06	440	47	11	0.15	0.01	0.03	0.5	0.52	2	0.29	0.76	0.08	0.06
EKS10	40	3.56	0.42	6.2	0.04	457	174	1	0.06	0.05	0.56	3	0.2	1	6.62	1.07	0.12	0.19
EKS11	10	0.38	0.03	0	1.59	440	29	418	0.2	0.25	0.07	0.31	0.27	66	0.03	0.76	0.08	0.23
EKS11	60	0.11	0.01	0.09	0.03	440	82	27	0.14	0.01	0.01	0.1	7.88	9	0.1	0.76	0.09	0.10
EKS11	75	0.11	0.02	0.16	0.01	443	145	9	0.13	0	0.02	0.09	8.75	0	0.18	0.81	0.18	0.22
AVERAGE	32	0.70	0.09	0.69	0.19	440.63	76.47	49.21	0.12	0.06	0.08	0.62	3.65	16.36	0.78	0.77	0.15	0.13

Where: **TOC:** Total Organic Carbon, TOC (%) = PC + RC; **S1**: quantity of free hydrocarbons (gas + oil), in mg/g of rock; **S2**: Quantity of thermally generated (cracked) hydrocarbons, in mg/g of rock; S3: Quantity of CO2 generated during pyrolysis of the sample, in mg/g of rock; **Tmax:** Temperature in °C, at which the largest quantity of hydrocarbons is released upon cracking; **PI:** Production Index; PI = S1/ (S1+S2); **PC**: Pyrolysable carbon Quantity PC = 0.083 X (S1 +S2); **RC:** Residual Carbon; **HI**: Hydrogen Index in mg/g of rock, HI = (S2*100)/TOC; **OI:** Oxygen Index in mg/g of rock, OI = (S3*100)/TOC; **GP**: Genetic Potential, GP = S1 + S2; **MinC:** Mineral Carbon; **S3CO:** Corresponds to the amount of CO released; **OICO**: Corresponds to the quantity of pyrolyzed CO relative to TOC, i.e. S3CO/TOC.

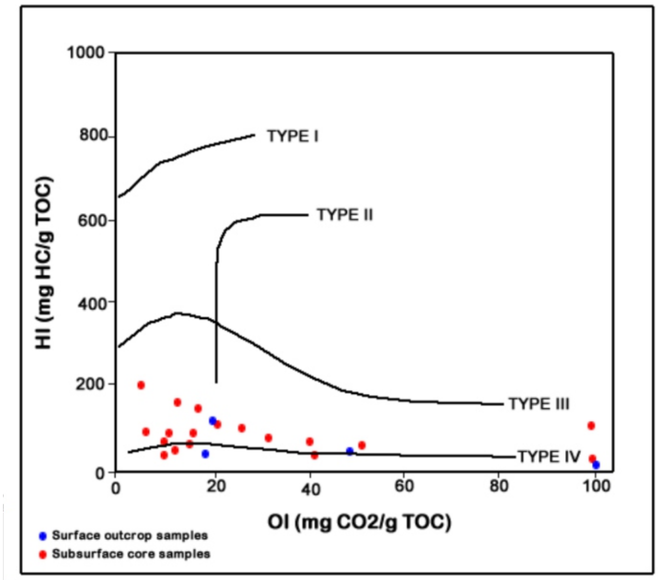


Figure 9: Modified van Krevelen diagram indicating the kerogen type of Ekenkpon Shale (After Hunt, 1995).

between petroleum generation and temperature. It can be estimated using results from vitrinite reflectance, maximum temperature of pyrolysis (Tmax) range, Numerical maturity calculation method and plotting Tmax versus HI and using the data to characterize the extent of thermal maturity of source rocks. However, it has been shown that there exists a linear relationship between Tmax and calculated Ro (0.018) *Tmax – 7.16); this was used to estimate the vitrinite reflection regimes of the samples (Jarvie and Lundell, 1991; Jarvie *et. al.,* 2007; Dembicki, 2009). The Tmax values of the studied area ranges from 429 to 4570C (av. 440.630C), the Ekenkpon Shale is marginal mature - mature for hydrocarbon generation (Figure 10a). Calculated Vitrinite reflectance values using the Tmax data, using the formula proposed by Jarvie et. al., (2007) range from 0.56 - 1.07% Ro (av. 0.77% Ro). this suggest that the source rock is mature and fall within the oil and gas window and indicates a good level of thermal maturity, with high dry gas potentials. The plot relating depth and stages of generation of hydrocarbon by Hall *et. al.,* (2008) for the analysed samples from Ekenkpon Shales corroborates the fact that it has limited ability for generating liquid hydrocarbon (Figure 10b) and enormous potential for dry gas (CH_4). From the initial studies of Espitalie (1986), he suggested a boundary between immature and mature kerogen (oil production zone) for Type III organic matter corresponding to a Tmax value of 434°C, while Tmax value of 465oC connotes the boundary between mature and over-mature kerogen (gas-production zone). The mean Tmax values of the analysed samples is 440.63°C, with a maximum Tmax value of 457°C was recorded for samples from well EKS10 at 40m (Figure 10c). The analysis of all the critical Rock-Eval parameters (HI, OI and Tmax) refers to early maturity to peak maturity level for the analysed Ekenkpon Shales

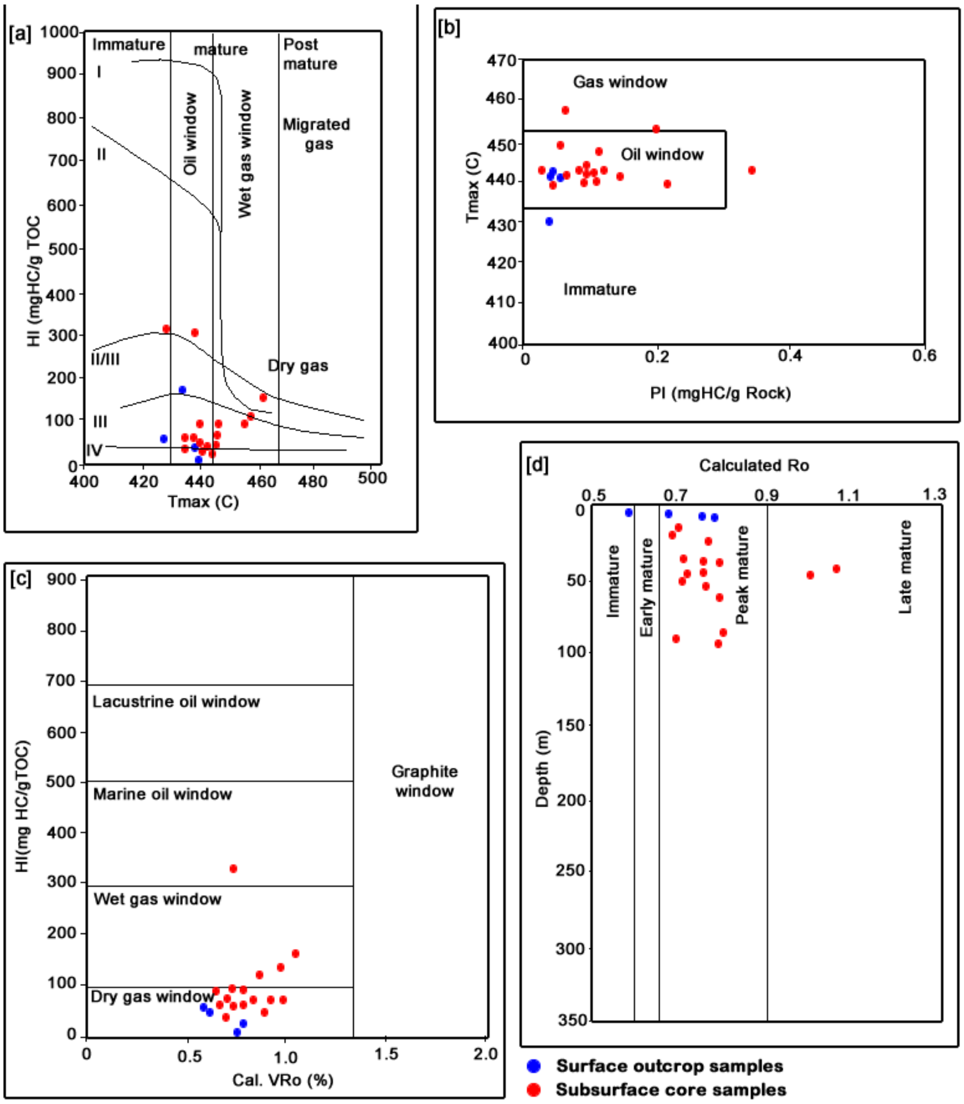


Figure 10: a - HI versus Tmax cross plot. The plot shows the kerogen type and maturity range of the samples (fields after Espitalie, 1986); b - Tmax versus Production Index (Ghori, 2002). c - Depth versus calculated maturity of Ekenkpon Shales (After Peters and Cassa, 1994). d - HI vs. calculated VRo plot of the studied samples (after Dembicki, 2009).

samples with an average vitrinite reflectance of 0.77 R0 showing peak maturity (Figure 10d).

Migrated Hydrocarbons

The comparison of production indices with the thermal maturity stage of samples was used to identify migrated hydrocarbons (Hunt, 1996). High S_1 values are either normal, which indicate potential source rocks; or abnormal, resulting from a combination with migrated oil, or coming from drilling additives (Peters and Cassa, 1994). When S_1 is high and the TOC is low, nonindigenous hydrocarbons can be detected (Hunt, 1996). Figure 48 is used to differentiate migrated from non-migrated hydrocarbons for the analysed samples in different boreholes. The dividing line on the plot is where S_1/TOC = 1.5. Values belonging to nonindigenous hydrocarbons appear above this line while indigenous hydrocarbon values emerge below it (Hunt, 1996). The cross plot of S_1 versus TOC (wt.%) was used to distinguish migrated hydrocarbons and contaminants from indigenous hydrocarbons. Figure 11 represents the plot of S_1 versus TOC for the analysed samples in this study. From the plot, the organic matter in the analysed Ekenkpon Shales are indigenous in origin.

The organic geochemistry analyses show that the Ekenkpon Shales are having TOC ranges from 0.03 to 4.15 wt.% with an average of 1.43 wt.% and thermally mature, consists of mainly Kerogen Type III, deposited in anoxic condition and mature. The Ekenkpon Shale has fair to good source rock generative potential and has obtained thermal maturity levels equivalent to the oil and

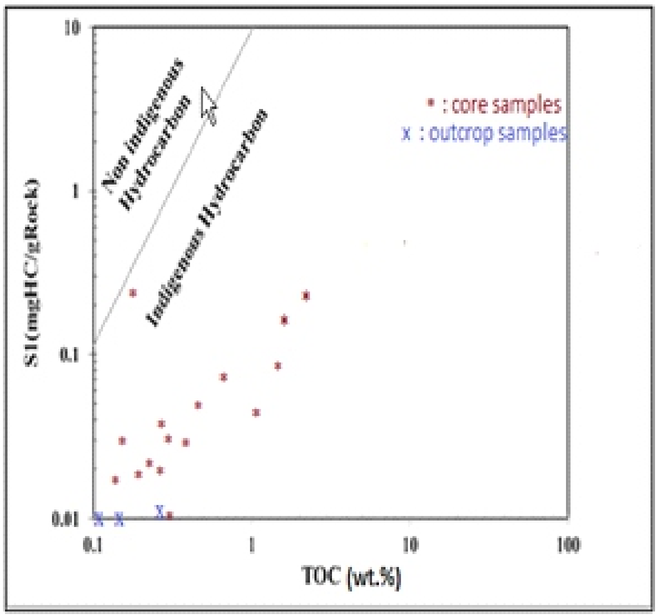


Figure 11: S_1 versus TOC (wt.%) cross plot shows all the samples are in the zone of indigenous hydrocarbon (after Hunt, 1996).

wet gas window. The thermal maturity of the shales is controlled by the burial history of the sediments. The Ekenkpon Shale have good prospects for shale gas exploration.

Mineralogical Composition analysis

The result of XRF analysis is presented in Table 3. Of significance in shale gas exploration is the mineralogical content of the shale and its quantity of organic matter (TOC). XRF analysis enabled the characterization of the mineral phases in the samples and using the normative methods, the various clay mineral constituents using the Paktunc (2001) method. The result shows the abundance of illite followed by kaolinite. The normative minerals were determined from the XRF elemental analysis using computer algorithm using the methodology of Cohen & Ward (1991). The goal of normative analysis is to determine the mineralogy of rocks from their bulk chemical composition. The XRF analysis results shows an average mineral composition of Ekenkpon Shale as quartz (25.75%), other carbonates (11.66), illite (33.64%), chlorite (9.06%), Kaolinite (16.22%), pyrite (1.39%), apatite (1.28%), rutile (0.96%) and halite (0.04). This is an indication of high silica, moderately low carbonate and hence points to the ease with which the shale may be amenable to fracking under hydraulic pressure. The XRF derived Ekenkpon shale mineralogy data are summarized below. The results are represented as pie chart for easy interpretation (Figure 12).

MINERAL	RANGE (%)	AVERAGE (%)
Quartz	9.49 – 36.57	25.75
Carbonates	0 – 76.12	11.66
Illite	11.28–53.24	33.64
Chlorite	0 – 29.6	9.06
Kaolinite	1.12 – 42.74	16.22
Pyrite	0.10 - 10.87	1.39
Apatite	0 - 3 1.9	1.28
Rutile	0.28 – 1.73	0.96
Halite	0 – 0.16	0.04

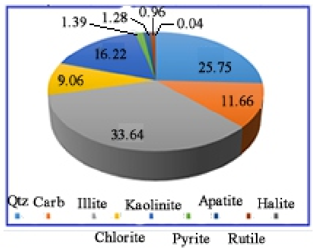
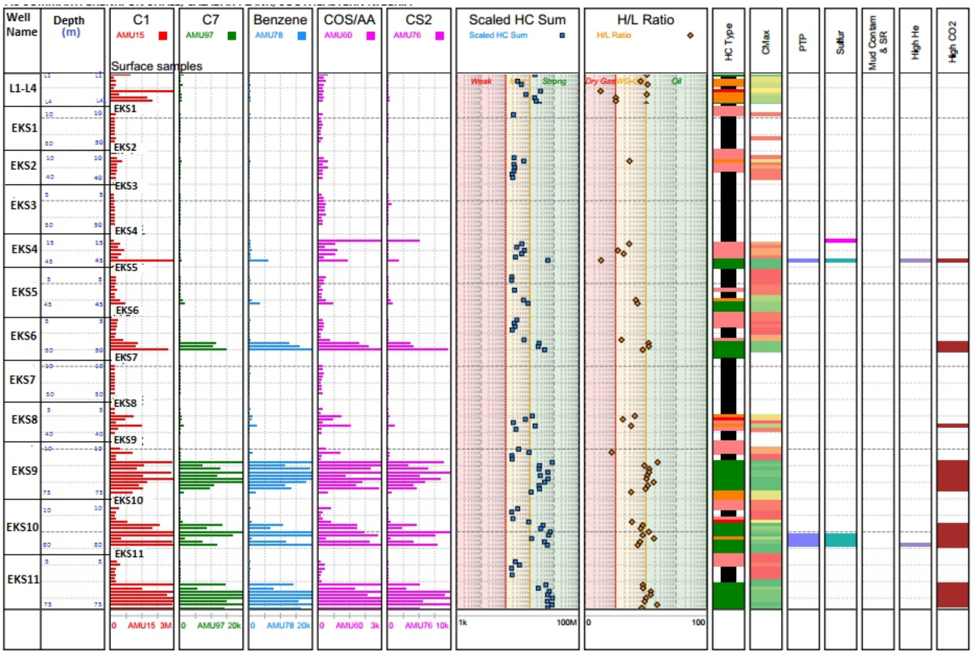


Figure 12: Diagrammatic representation of mineralogical analysis result.

The XRF results reflects clay, carbonates and silica as the dominant minerals in all samples across all depths. The distribution of illite and kaolinite within the samples may be used as paleoclimatic indicator during deposition. The XRF analysis demonstrate that the samples generally contain more than one-third clay minerals. Detrital quartz occurs as silt size grain. Organic materials are present in the form of flakes or sub rounded particles and scattered over the rock matrix. Under the microscope, silt size quart grains predominate with abundant organic matter, mica and pyrite grains. Presence of fine grain pyrite nodules in shales indicates deposition within an anoxic environment which supports high organic matter preservation (Shen *et. al.,* 2007). Also, the occurrence of pyrite in the Ekenkpon Shales shows that the shales were deposited under an anoxic sedimentary environment where reduced iron (Fe_2+) can exist in solution.

Fluid Inclusion Studies (FIS)

Petroleum fluid inclusions can be found along hydrocarbon migration pathways within present day petroleum reservoirs and within exhumed and flushed petroleum reservoirs. The identification and study of petroleum inclusions has thus far normally been accomplished through optical petrography of doubly-polished thin sections. Although these methods are important to any inclusion study, relying on this approach alone requires that thin sections be prepared from each zone of interest. It provides only the most basic chemical information on the trapped fluid phase, and it cannot be applied easily to dry gas inclusions because they do not fluoresce. Improved technology in the application of fluid inclusion stratigraphy provide information about the composition and conditions of entrapment of past fluids, including hydrocarbons. FIS has high level sensitivity and speed allowing detection of petroleum in samples that are well beyond the reach of standard GCMS methodologies (Hadley *et. al.,* 1998). The results of the FIS analysis for characterizing the fluid contents are summarized per well in Figure 13. Each spectrum is indexed according to depth and indicates the log of the millivolt response for atomic mass units (AMU) or mass to charge ratio (m/z) from $2 \leq m/z \leq 180$ and ratios. There is high level of dry gas (C_1 or CH_4) from the FIS species in most of the wells and increasing benzene content



FIS Interpretation Legend

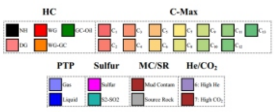


Figure 13: FIS Summary for Ekenkpon Shale Highlighting Major Fluid Contents.

Table 4: List of the organic molecules routinely reported by Fluid Inclusion Stratigraphy analysis and their mass fragments (After Barclays et. al., 2000).

[illegible]	[illegible] name	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	H_2S	Sour gas risk, ± bacterial alteration
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible] + alkanes)	[illegible] indicator
[illegible]	[illegible]	[illegible]
[illegible]	C_5^+, S_2 and SO_2	[illegible]
[illegible]	[illegible] + alkanes	[illegible]
[illegible]	[illegible] and C_6^+ hydrocarbons	[illegible]
[illegible]	C_6^+ aromatics	[illegible]
[illegible]	[illegible]	[illegible]-type indicator
[illegible]	[illegible]	[illegible]-type indicator
[illegible]	[illegible] $C_7H_{15}^+$)	[illegible]-rich inclusions
[illegible]	[illegible]	[illegible] -maturity gases
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible] gas (wet gas + cyclic alkanes)	[illegible] -water transition zone, oil composition or biodegradation
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible] pay
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible] inclusion indicator
[illegible]	[illegible] range alkylated cyclic alkanes + toluene)	[illegible]

indicating proximity to pay at deeper depths. Table 4 below among other indices like the pixler ratios, wetness and balance are applied for effective FIS interpretations. Nine wells show high dry gas potential with limited liquid hydrocarbon and condensates while two wells are completely dry wells (water bearing).

DISCUSSIONS

During routine hydrocarbon exploration, the stratigraphic location of source beds and their reservoir rocks is key in evaluating for the presence of any significant play zone. This is further enhanced where all the hydrocarbon elements are in-place (timing) before the structuration the builds up the trapping system and then hydrocarbon maturation. This study reveals light grey to dark grey or black, predominantly carbonaceous and siliceous shales making up the Ekenkpon Shale and suggests abundant inputs of organic matter and the silica content derived from terrigenous materials (quartz). The quality of kerogen (Type III) is predicated upon the abundance of higher plant remains and continental origin of majority of the organic matter. The Ekenkpon Shale resulted from the second transgressive phase that ensued during the stratigraphic evolution of the Calabar Flank with abundant ammonites and marine foraminifera microfossils. Therefore, the terrigenous input in its constituents alludes to contribution of materials from surrounding environments and an extensive period during deposition. The laminations reflect sedimentation process within a quiet/still condition, indicating changes in sediment influx and chemical conditions (Eh & pH) of the basin, along with differences in the rate of sedimentation. The need for a high-resolution stratigraphic framework for the Ekenkpon Shale is over emphasized. More recently, chemostratigraphic principles have been integrated with conventional biostratigraphic and lithostratigraphic analysis in characterizing shale gas plays (Ratcliffe et. al., 2011, Ratcliffe and Wright 2012). Mineralogically, the Ekenkpon shales is made of quartz, carbonates, micas, lithic grains and Fe sulphide (pyrites); having silt size grains in an argillaceous matrix. On the average, clay minerals constitute the bulk of the mineralogical composition with illite, kaolinite and chlorite amounting to about 68.45%. Silica (av. quartz = 25.75%) and carbonate (av. 11.66%) and subordinate heavy minerals constitute the rest. The grains are sub-rounded to well-rounded suggesting that the sediments have been subjected to long distance transportation or multi-cycles of deposition. The existence of kaolinite is an indication that low pH condition prevailed during the deposition and active chemical weathering environment under humid and suggests tropical climate depending largely on the on the source (parent) rock constituents. However, because kaolinite is stabled at low pH (and shallow depth), the abundance of illite (abundant with increasing depth) points to higher maturity of the sediments (Weaver, 1989). Kaolinite, illite and chlorites are the major clay components of the Ekenkpon Shale with little or no montmorillonite as indicated from the XRF mineralogical studies. The presence of Iron sulphide (pyrite - FeS_2) points to more of reducing environment. The inorganic geochemical characteristics of the shale unit and other physical sedimentological features (more brittle in nature) are important for the frackability of the shale when subjected to hydraulic fracturing. Organic geochemical analysis indicates the presence of early mature - mature gas-prone source rock in most of the samples. Although not in abundance, the presence of phosphorus is indicative of near-shore upwelling environments with high surface productivity. The dark grey to black shales deposited in alternation between oxic and anoxic depositional conditions is responsible for the variation in TOC (0.03 to 4.15) wt % of the analysed samples. Evaluation of the thermal regime of the shale confirms fair to good genetic potentials and suggest that it's been through considerable thermal cracking capable of producing oil, condensates and gas especially as observed from finding of fluid inclusion studies (studies point to essentially gas prone OM). Because the study of the shale gas potential of the Ekenkpon Shale is still in its infancy, the results obtained from analysis were compared with analogous gas producing shales worldwide (Table 5).

Extensive studies on the US commercial gas shales (Barnett, Woodford, Marcellus, Fayetteville, Haynesville) shows that the shales have appreciable and comparable TOC values (1 to 25 wt %) vitrinite reflectance (VRo = 0.7 to 3.8%) pointing to mature deposits. This implies that the geochemical properties of Ekenkpon shales is very similar to those of other commercial gas shales deposits. Considering the depth factor, the Antrim Shale (Michigan Basin) and New Albany Shale (Southern Indiana and Northern Kentucky) are producing gas from shallow depth. The Antrim Shales gas is of both thermogenic and biogenic origin with 0.4 to 0.6% VRo while producing from the depth of 350 to 790m. The New Albany Shale have 0.4 to 0.6% VRo and producing from 160 to 600m depth (Table 6). The depth and thickness indications of Ekenkpon Shale could not be ascertained due to the shallow wells used for this study, however data from previously drilled wells (Ituk-2 and Anua-1) shows the thickness of Ekenkpon shale between 580-817 m (Odumodu, 2012). The limitation in this contribution stems from the premise that the samples analysed were derived from shallow exploratory wells in the Ekenkpon Shale, hence, stratigraphically deep sections were not attained and the true thickness of the shale unit was not ascertained. Although the Ekenkpon shale is tight, with low permeability, modern technology may be utilized such as horizontal drilling and multi stage hydraulic fracturing to improve the permeability near the

Table 5: Comparison of Ekenkpon Shale Properties with analogous producing gas shales (Compiled from Slatt and Rodriguez, 2012; Jarvie et. al., 2007; Liu et. al., 2013).

Shale	Basin	Depth (m)	Thickness (m)	TOC wt.%	Maturity VR_o%	Quartz %	Clay %	Kerogen Type	Age
Barnett	Fort Worth	1828-2743	60-155	3.0-8.0	1.2-2.0	40-60	33	II & III	Upper Mississippian
Woodword	Andarko	1828-4227	20-100	3.0-10.0	1.1-3.0	60-80	20-45	III	Devonian
Marcellus	Appalachian	465-790	20-100	2.0-10.0	0.6-3.0	40-60	33	II & III	Lower Devonian
Fayetteville	Appalachian	457-1981	20-110	4.0-10.0	1.2-4.0	40-60	20-60	III	Upper Mississippian
Haynesville	Arkansas	3048-4267	50-110	0.5-8.0	1.5	<40	20-60	III	Jurassic
Antrim	Michigan	350-1000	100-305	1.0-25.0	0.78-1.2	<40	<40	II	Late Devonian
New Albany	Illionis	160-600	100-300	0.5-8.0	0.4-0.6	<40	20-50	III	Devonian & Mississippian
Barren Measures	Raniganj	70-1200	80-800	3.75-20.9	0.6-1.1	49.02	20.69	III	Late Permian
Ekenkpon	Calabar Flank	0-2922?	50-817?	0.03- 4.15	0.6-1.0	25.75	33-68	III	Cenomanian-Turonian

well bore.

The source rock can generate gas; however, the present temperature and depth level is not necessarily an indicator of its maturity. Maximum burial and required temperature have occurred in the geological past. The evaluation of reservoir characteristics from fluid inclusion stratigraphy include the fluid contents and petrophysical properties of rocks from megascopic study showing fracture fabrics. Most of the pores are meso-pores and associated with organic matter and clay matrix. The shales are highly heterogeneous, moderate to well sorted and fractured. Fluid inclusion stratigraphy shows that Ekenkpon shale shows high potential for dry and wet gas. Also, there are visible evidence of gas condensates and oil stains at the deeper sections. The high content of quartz (avg. 25.75%) and high carbonates (11.66%) contents in the Ekenkpon Shale, make the shale to be more brittle in nature and this may help to stimulate more fractures during artificial hydraulic fracturing. The brittleness index was calculated from the XRF and fluid inclusion studies. This view is supported from the classical example of gas producing Barnett Shale which is producing gas from the lithologic units having 45% quartz and 27% clay (Bowker, 2007). The similarity in the mineralogy is observed in other shales of New Albany, Marcellus, Woodford, Fayetteville, Antrim, etc.

SUMMARY AND CONCLUSIONS

This study is an effort to highlight the mineral composition, organic richness, and maturity in order to evaluate the gas generation potential and reservoir quality of Ekenkpon Shale. Integrated studies were utilized to understand the mineralogical, geochemical and petrophysical characteristics of Ekenkpon Shale to validate the shale gas prospects in Calabar Flank. The shales have fair to good source rock generative potential and have reached thermal maturity levels equivalent to the oil window. The main hydrocarbon product expected from Ekenkpon Shales is mainly gas as the organic matter are derived from type III kerogen, thus a good prospect for shale gas exploration. There are also prospects for oil and condensates from analysis of fluid inclusion data.

In conclusion, the Ekenkpon Shales has favourable mineralogy, geochemistry and reservoir characteristics to qualify it as a potential shale gas play. The shale properties are similar to US commercial gas shales. Moreover, the micro scale fabric anisotropy and brittleness of the shale supports the development of variable fracture networks, which can boost the artificial hydraulic fracturing. As the maturity and FIS species is found to increase with depth, the gas window can be expected at greater depth. Hence, shale gas exploration strategies should focus more on the deeper sections as the conclusions presented here are only preliminary.

RECOMMENDATIONS

It is highly recommended that efforts towards drilling deeper wells in the Calabar flank with the intent of exploring for its shale gas potentials. This approach will enable characterization of the geochemistry and fluid content of these currently unassessed zones.

Also, before any final conclusion; several geophysical well logs should be run to delineate organic rich shale facies, its porosity, permeability and bulk density of the Ekenkpon Shale.

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