Source Rock Evaluation of Shale in Parts of Ini Local Government Area, Akwa Ibom State, Southeastern Niger Delta Basin, Nigeria

Ekpedeme R. Asuaiko, Clement E. Bassey, Thomas A. Harry, Tejumade A. Owoeye and Veronica E. Ibanga

ABSTRACT

Total organic carbon and Rock – Eval pyrolysis studies were conducted on seven (7) shale samples outcropping in parts of Ini Local Government Area of Akwa Ibom State, Southeastern Niger Delta, Nigeria. The studies were done to determine the quantity and quality of organic matter in the shale source rock, and their hydrocarbon generation potential. Total organic carbon (TOC) values indicate poor to excellent organic richness with values ranging from 0.46 wt. % to 5.98 wt. %. The free oil content (Sn) values range from 0.02-0.21 mg HC/g rock, (mean = 0.07 mg HC/g rock), while the source rock potential (S2) ranges from 0.08-1.09 mg HC/g rock with an average value of 0.45 mg HC/g rock both indicating poor source rock generative potential. The Hydrogen Index (HI) and the Oxygen Index (OI) range from 11 mg HC/g TOC to 59 mg HC/g TOC and 41 mg CO/g TOC to 74 mg CO/g TOC with an average value of 27.28 mg HC/g TOC and 55.71 mg CO/g TOC respectively. HI versus OI and S2 versus TOC cross plot results indicate that shale in parts of Ini Local Government Area contains organic matter capable of generating kerogen type III to type IV which is gas prone.

Keywords: Kerogen Type, Organic matter, Rock-Eval Pyrolysis, Shale.

I. INTRODUCTION

One of the components of a petroleum system is a source rock, without which a petroleum play does not exist, and without which other components and processes are immaterial. Source rocks are fine–grained, water–deposited sedimentary rocks such as shale, limestone and mudstone that contain abundant quantities of organic matter to generate and expel economic quantities of oil and or gas when heated [1]-[6].

Deposition of source rocks requires the existence of specific environmental conditions to aid biological productivity which, in turn, determines the amount of organic matter preserved in the sediments. Organic matter that are preserved in sediments include humic material, resins, waxes, and lipids. Environments that conserve unusually large amounts of organic matter are large, stagnant water areas and silled basins, where the bottom waters, owing to anoxia, are strongly reducing, with organic contents in sediment frequently exceeding 15.0 wt. % [7].

The potential of a source rock to generate petroleum (oil and or gas) is closely linked to the quantity, quality, and thermal maturity of organic matter present in the rock. In this research, total organic (TOC) and Rock–Eval pyrolysis studies were conducted on shale outcropping in parts of Ini Local Government Area, Southeastern Niger Delta, Nigeria to evaluate the quantity and quality (kerogen type) of organic matter available in the shale, as well as evaluate the type of kerogen in order to establish whether it is suitable to generate either oil, gas or both. The result will provide additional information required to advance development in petroleum exploration and exploitation in this part of the Niger Delta.

II. GEOLOGY OF THE STUDY AREA

The study area extends between latitudes 5º 18' - 5º 30' N and longitudes 7º 37' - 7º 52’ E. It is found in the south – eastern part of the Niger Delta which is located in the Gulf of Guinea. The lithostratigraphic units in the subsurface of the basin are the Akata Shales Formation, Agbada Formation, and the Benin Sands Formation. Their outcropping units
include the Imo Formation and the Ameki Group consisting of Amekei, Nanka, Nsugbe and Ogwashi – Asaba Formations [8]. The study area is underlain by the Imo Formation which consists of thick clayey shale, fine textured dark grey to bluish grey shale and thin sandstone bands. It consists of two main rock types which are clay/shale and sandstone [9]. The sandstone at Odoro – Ikpe and Ikporom are massive. The sandstone at Ikporom is distinguished by flaser beds(fine laminations and streaks of white clay). Texturally, it is fine to very fine grained whereas the one at Odoro-Ikpe is coarse to very coarse grained strata. However, [10] records that geologically, the dominant characteristic is sandstones and limestones east of Nkaribi and Obotme. Also, [11] affirms that the study area mainly composed of the Benin Formation with Bende – Amekei Group and Imo Shale Formation exposed in a few parts. Fig. 1 shows the study area superimposed on the geology of the area, extracted from the geologic map of Akwa Ibom State, Nigeria.

III. MATERIALS AND METHODS

Shale samples were the main experimental materials. Samples were collected from outcrops in parts of Ini Local Government Area, Akwa Ibom State, Southeastern Niger Delta, Nigeria, located between Latitudes 5° 19’ 00” - 5° 31’ 00” N and Longitudes 7° 42’ 00” - 7° 51’ 53” E. Global Positioning System was used to establish sample locations. Sampling was done by pitting with a shovel to a depth of about 50 cm (0.5m). Each of the collected samples was sealed up in a zipper storage bag and labeled using a masking tape and a permanent marker. The samples were later air dried and seven (7) out of 15 samples were pulverized. 100 mg was measured out of each pulverized sample. Each weighed sample was packed in a small zipper storage bag. The seven samples, packed in a bigger zipper storage bag, were shipped through courier to the laboratory of GeoMark Research Limited in Houston, Texas, USA for geochemical analysis.

To determine the organic richness and organic matter type, the shale samples were analyzed using the methods and procedures outlined by [1]-[5], [12]-[16]. Total organic carbon was determined using LECO C230 carbon analyzer while Rock Eval pyrolysis was done using a Rock - Eval 6 analytical instrument.

IV. RESULTS

The geochemical analyses for seven (7) shale samples from outcrops in some parts of Ini Local Government Area of Akwa Ibom State, Southeastern Niger Delta, Nigeria have been interpreted. The total organic carbon (TOC) and the rock–eval pyrolysis results and other useful parameters are presented in Table I. Source rock evaluation is based on the appraisal of several factors [5]. TOC is a measurement of the organic richness of sedimentary rocks [17]. The shales as shown in Table I have TOC values ranging from 0.46 wt. % to 5.98 wt. % with a mean value of 1.65 wt. %. The mean value surpasses the TOC threshold value of 0.5 wt. % required for sediment to generate hydrocarbon [1], [3], [17]-[19]. A plot of TOC versus depth (sample identification numbers since samples were collected from field outcrops) is displayed as Fig. 2.

TABLE I: TOC AND ROCK EVA L PYROLYSIS RESULTS AND OTHER DERIVED PARAMETERS

Fig. 1. Geology of the Study Area.

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OF THE STUDIED SHALE OUTCROP SAMPLES FROM PARTS OF INI L.G.A.

| Sample ID | % Carbonate (% w/w) | TOC (wt%) | S1 (mg HC/g) | S2 (mg HC/g) | S3 (mg CO2/g) | Tmax (°C) | Calc. % R0 (RE TMAX) | HI (S2×100/TOC) | OI (S3×100/TOC) | S2/S3 | S1/TOC | PI (S1/(S1+S2)) | GP S1+S2 |
|-----------|---------------------|----------|--------------|--------------|--------------|-----------|---------------------|----------------|----------------|--------|---------|----------------|---------|
| Sample 1  | 12.03               | 1.69     | 0.09         | 1.00         | 0.69         | 398       | 0.00                | 59             | 41             | 1      | 5       | 0.08           | 1.09    |
| Sample 2  | 13.30               | 1.51     | 0.08         | 0.68         | 0.62         | 394       | 0.00                | 45             | 41             | 1      | 5       | 0.11           | 0.76    |
| Sample 3  | 11.25               | 0.62     | 0.03         | 0.15         | 0.46         | 414       | 0.29                | 24             | 74             | 0      | 5       | 0.17           | 0.18    |
| Sample 4  | 12.49               | 0.46     | 0.02         | 0.08         | 0.27         | 427       | 0.53                | 17             | 59             | 0      | 4       | 0.20           | 0.10    |
| Sample 5  | 18.54               | 5.98     | 0.21         | 1.09         | 4.41         | 353       | 0.00                | 18             | 74             | 0      | 4       | 0.16           | 1.30    |
| Sample 6  | 14.94               | 0.72     | 0.04         | 0.08         | 0.38         | 413       | 0.27                | 11             | 53             | 0      | 6       | 0.33           | 0.12    |
| Sample 7  | 15.93               | 0.58     | 0.03         | 0.10         | 0.28         | 425       | 0.49                | 17             | 48             | 0      | 5       | 0.23           | 0.13    |

The basic parameters obtained from rock–eval pyrolysis after [1] and [2] include S1, which is the amount of the free hydrocarbon content in the sample.

The S1 values vary from 0.02 mg HC/g to 0.21 mg HC/g as displayed in Table I, with an average value of 0.07 mg HC/g.

The S2 values of the samples, representing the quality of hydrocarbons generated through thermal cracking of kerogen, from Table I range from 0.08 mg HC/g to 1.09 mg HC/g with a mean value of 0.45 mg HC/g. These values are low and show poor source rock potential as indicated in Fig. 3 which is plot of depth (sample identification numbers since samples were collected from field outcrops) versus oil potential.

The S3 values, indicating the amount of CO2 produced during pyrolysis of kerogen, vary from 0.27 mg CO2/g to 4.41 mg CO2/g as shown in Table I, with an average value of 1.0 mg CO2/g.

The oxygen index (OI), which is the parameter that correlates the ratio of the oxygen to carbon in the sediment, was calculated from S3 values. The OI values vary from 41 mg CO/g TOC to 74 mg CO/g TOC, averaging 55.71 mg CO/g TOC.

From the rock–eval pyrolysis data, organic matter type in hydrocarbon source was characterized and presented as hydrogen index (HI) which evaluates the origin of organic matter. HI values vary from 11 mg HC/g TOC to 59 mg HC/g TOC, averaging 27.28 mg HC/g TOC. According to [3], these values indicate gas prone sediment as confirmed by the plot in Fig. 4 which is plot of depth (sample identification numbers since samples were collected from field outcrops) against HI.
The values of Production Index (PI), as shown in Table I range from 0.08 to 0.33, averaging 0.18. These values are used to characterize the evolution of organic matter [5]. The plot of PI versus depth (sample identification numbers since samples were collected from field outcrops) as presented in Fig. 5 shows that shale samples span from immature, non-generative to gas prone source rock.

![Plot of Depth against Production Index](image)

**Fig. 5: Plot of Depth against Production Index.**

### V. DISCUSSION OF RESULTS

#### A. Quantity of Organic Matter

Organic matter includes both soluble bitumen and insoluble kerogen both being represented by total organic carbon (TOC) readings. Sufficient amount of organic matter is an essential prerequisite for sediment to generate oil or gas [20]. Organic matter quantity (richness) of shales in parts of Ini Local Government Area of Akwa Ibom State, Southeastern Niger Delta, Nigeria, was evaluated based on TOC contents [2]-[4]. The TOC and Rock–Eval analytical data performed on seven (7) shale samples from field outcrops in the study area are summarized in Table I. The TOC values vary from 0.46 to 5.98 wt. % with a mean of 1.65 wt. % which exceeds the minimum threshold of 0.5 wt. % required for source rocks to generate oil or gas [1]-[3] and [18]. Reference [3] proposed that a TOC content of 0.5 to 0.5 wt. % is indicative of a poor source rock, 0.5 to 1.0 wt. % indicates fair source rock, 1.0 to 2.0 wt. % indicates good source rock, 2 to 4 wt. % indicates very good source rock and a TOC value greater than 4 wt.% shows an excellent source rock. The TOC values of the analyzed shale samples reveal a source rock that range from poor to excellent organic carbon content with TOC values ranging from 0.46 to 5.98 wt. % (Fig. 2). Sample 5 exhibits an excellent source rock potential with a TOC of 5.98 wt. % as against sample 4 which shows a poor source potential with a TOC of 0.48 wt. % (Table I). Samples that have values between 0.5 and 1 wt. % are slightly rich of organic matter and potentially fair source rocks.

#### B. Quality of Organic Matter (Kerogen Type)

The type of organic matter present in source rocks determines the quality, and kerogen composition determines the petroleum potential. Organic matter quality refers to whether organic matter is suitable to generate either oil, gas, or both [1] and [21]. Rock–Eval pyrolysis S2 and S3 data were used to characterize the organic matter type (kerogen type). These data were presented as hydrogen index (HI = \( S_2/TOC \times 100 \)) and oxygen index (OI = \( S_3/TOC \times 100 \)) (Table I). A Pseudo van Krevelen diagram [22] showing a plot of HI versus OI reveals a Type III to Type IV kerogen (Fig. 6). This is characterized by a low HI values ranging between 11 and 59 mg HC/g TOC and a fairly high OI values ranging from 41 to 74 mg CO2/g TOC, suggesting a terrestrial origin of organic matter composed of woody or herbaceous plants.

![Plot of HI versus OI, determining the type of kerogen present in the source rock](image)

**Fig. 6: Plot of HI versus OI, determining the type of kerogen present in the source rock.**

A cross plot of S2, that is, remaining hydrocarbon potential against TOC describing kerogen type, was also used to evaluate the organic matter in shales in the study area [23] and [19]. It reveals that the source rock contains mostly Type III to Type IV kerogen and are dry gas prone as displayed in Fig. 7. According to [4], hydrogen content of organic matter is the most important factor controlling the type of hydrocarbon generated by a source rock. Pyrolysis HI values are commonly used because this parameter is difficult to determine. Reference [3] proposed that HI for non-generative Type IV kerogen is less than 50 mg HC/g TOC, gas prone Type III kerogen is between 50-200 mg HC/g TOC, mixed oil and gas prone type II/III kerogen is between 200-300 mg HC/g TOC, oil prone Type II kerogen
is from 300 to 600 mg HC/g TOC whereas for oil prone kerogen Type 1, HI is greater than 600 mg HC/g TOC. From Table I, the HI values are low, varying between 11 and 59 mg HC/g TOC, mostly indicating non–generative to gas prone source rock.

Kerogen type interpretation is further supported by the hydrocarbon type index (S2/S3), which indicates the potential of the source rock to generate hydrocarbon (gas or oil or both). A source rock with hydrocarbon type index of less than 1 indicates Type IV kerogen and non–generative potential, 1–5 indicates Type III kerogen and gas generation potential, 5–10 shows TypeII/III kerogen and mixed oil and gas potential, 10–15 indicates Type II kerogen and oil generation potential while a hydrocarbon index value of greater than 15 indicates Type I kerogen and oil generation potential [3]. Considering the ratio of S2/S3 of the studied samples as presented in Table I, which are between 0 and 1 mg HC/mg CO2, samples 1 and 2 have Type III kerogen with gas generation potential while samples 3 to 7 have Type IV kerogen with non–generative potential. Therefore, the kerogen products are non–generative to gas dominant.

Fig. 7. S2 versus TOC cross - Plot to Identify Kerogen Type.

The temperature, T_max, is influenced by the type of organic matter during diagenetic stage and the beginning of catagenesis. It is lower in the terrestrial kerogen Type III and higher in the marine or lacustrine Types 1 and II[1]. From Table I, T_max values vary from 353 to 425°C. These values are low (< 430°C) indicating terrestrial origin of the organic matter.

C. Potential for Hydrocarbon Generation

Source rock potential for generation can be classified into poor, fair, good, very good and excellent depending on pyrolysis S2 and TOC values [3]. These values are displayed in Table II. Based on Table II, all but one analyzed sample has TOC content greater than 0.5 wt.% minimum requirements for source rock to generate hydrocarbon (Table I). Sample 4 shows a poor source rock potential with a TOC content of 0.46 wt.%, samples 3, 6 and 7 exhibit a fair potential with TOC content of 0.62, 0.72 and 0.58 wt. % respectively while sample 5 exhibits an excellent potential to generate hydrocarbon with a TOC content of 5.98 wt. %.

Thus, based on TOC, the analyzed samples show poor to excellent potential for hydrocarbon generation. Pyrolysis S2 values of the studied samples range from 0.08 mg HC/g to 1.09 mg HC/g (Table I). These values are low, and based on the assessment criteria presented in Table II, samples indicate poor source rock potential. Since the pyrolysis S2 yield and TOC values explain the quality and quantity of the organic matter present within the investigated samples, on the basis of these low values of S2 and TOC therefore, the examined samples can be considered as poor potential source rocks for the generation of hydrocarbon. This is in conformity with the previously observed HI values of 11–59 mg HC/g TOC and is consistent with Type III kerogen (Fig. 6).

The Genetic Potential (GP) is the sum of the values S1 and S2. It represents the amount of petroleum–oil and gas—that the kerogen is able to generate, if it is subjected to a sufficient temperature during an adequate interval of time. This potential depends on the nature and abundance of kerogen, which in turn are associated with the original organic input at the time of sediment deposition and to the conditions of microbial degradation and rearrangement of organic matter in the young sediment. [1]and [4] proposed that source rocks with GP less than 2, from 2 to 5, from 5 to 10 and >10 are considered to have poor, fair, good, and very good generation potential respectively. The GP values for the studied shale samples from outcrops in parts of Ini Local Government Area range from 0.10 to 1.30 mg HC/g rock with an average value of 0.53 mg HC/g of rock indicating poor generation potential. It is observed that GP values are closely correlated to TOC

Table II: Source Rock Generative Potential. After [3]

| Petroleum Potential | TOC (wt.%) | S1 | S2 |
|---------------------|-----------|----|----|
| Poor                | 0.0-0.5   | 0.0-0.5 | 0.0-2.5 |
| Fair                | 0.5-1.0   | 0.5-1.0 | 2.5-5.0 |
| Good                | 1.0-2.0   | 1.0-2.0 | 5.0-10.0 |
| Very Good           | >4.0      | >4.0   | >20  |
| Excellent           | >4.0      | >4.0   | >20  |
values (Table I). Samples with high TOC have high GP values. GP values are less than 2 mg HC/g rock corresponding to poor petroleum potential.

VI. CONCLUSION

A geochemical study was performed on seven (7) shale samples taken from field outcrops in parts of Ini Local Government Area of Akwa Ibom State, Southeastern Niger Delta, Nigeria. The investigation was done to assess the quantity and quality of the organic matter present in the samples, thermal maturity of the organic matter and their hydrocarbon generation potential. Total organic carbon values of the analyzed samples averaged 1.65 wt. %, Six of the analyzed samples have TOC of 1.69, 1.51, 0.62, 5.98, 0.72 and 0.58 wt. % placing them above the lower limit of TOC needed for a shale source rock. This is indicative of a good amount of organic matter in the sediment which suggests that shales in the study area are ‘sweet spots’ for some gas generation potential.

Pyrolysis S1 and S2 values for all the shale samples are relatively low denoting poor oil potential and thus signifying a poor source rock potential. S3 values, hydrogen index (HI) and production index (PI) all point towards gas prone sediments.

Rock – Eval data and the plot of HI and OI show that the shale samples are of a humic Type III to reworked /oxidized Type IV kerogen. Tmax and Production Index (PI) values signify that the samples are immature for hydrocarbon generation. The genetic potential of these samples is low and free hydrocarbons are absent. With the occurrence of Type III/IV kerogen, hydrocarbon yield and maturity levels, it is therefore concluded that the shale source rocks from parts of Ini Local Government Area have poor potential for oil generation. However, most of the shale samples have fair potential for gaseous hydrocarbons which has not been generated at the present outcrop level until the samples attain appropriate burial depth and thermal maturity. Similar geochemical study on Cretaceous outcrop samples of the Mamfe Basin yielded moderate to fair potential for gaseous hydrocarbon generation as a result of the prevalence of Type I11/Type IV kerogen. This is in agreement with our submission.

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