

Thermal Maturity and Hydrocarbon Generation Potential of Shales: A case study of Parts of Ini, Akwa Ibom State, Southeastern Niger Delta Basin, Nigeria

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Abstract

The thermal maturity and source rock potential of shale samples from outcrops in Ini, Akwa Ibom State, located within the Niger Delta Basin, Nigeria, were assessed through Total Organic Carbon (TOC), Rock-Eval pyrolysis, and vitrinite reflectance measurements. The TOC, Hydrogen Index (HI), and Genetic Potential (GP) exhibited a range of 0.46 to 5.98 wt %, with an average of 1.65 wt%. Additionally, HI values ranged from 11 to 59 mg HC/g TOC (mean = 27.28 mg HC/g TOC), while GP varied from 0.10 to 1.30 mg HC/g rock, averaging 0.53 mg HC/g rock. Classification based on Hydrogen Index (HI) versus Oxygen Index (OI) and S2 (represents the hydrocarbons generated from kerogen during pyrolysis) versus TOC plots identified the organic matter in the shales as Type III to Type IV kerogen. Furthermore, Tmax (oven temperature that corresponds with S_2) values spanning 353 to 427 °C, along with measured vitrinite reflectance values (Ro) of 0.37 to 0.38 %, indicated that the shales are thermally immature concerning hydrocarbon generation. HI versus Tmax and production index (PI) versus Tmax plots further supported this assessment of thermal immaturity in the shale samples.

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Keywords: Ini; hydrocarbon; thermal maturity; organic matter; source rock; vitrinite reflectance

1.0 Introduction

Source rocks are a vital element in a petroleum play, whether conventional or unconventional. They serve as precursors for the accumulation of hydrocarbons and contribute to reservoir potential (Egbobowaye, 2017). Source rocks include shale, mudstone, limestone, and marl. Hydrocarbons are generated in source rocks, which have abundant quantities of organic matter. The organic matter in source rock must be worked on by some groups of bacteria or buried deeply and heated in order to achieve the level of thermal maturity required to squeeze out hydrocarbons (Tissot & Welte, 1984; Hunt, 1996; Killops&Killops, 2005; McCarthy *et al.*, 2011).

Thermal maturity refers to the exposure of a source rock to heat over time. It involves diagenetic changes of organic matter from the immature phase to hydrocarbon generation and post maturity phases. These changes are propelled by temperature, time, and pressure. The level of thermal maturity increases with burial depth as a result of higher temperatures encountered in the deeper parts of the Earth's crust. It also depends on the time of exposure of source the rock to that temperature. Pressure mostly affects the physical properties of source rock, such as porosity and structural arrangement of particles (Hunt, 1996; McCarthy *et al*., 2011). The generation of petroleum from a source rock is attributed to the thermal transformation of organic matter.

Tissot & Welte, 1984; Peters & Cassa, 1994; Hunt, 1996; Killops & Killops, 2005; McCarthy *et al*., 2011 have discussed that hydrocarbon generation mechanisms differ from one basin to another depending on sedimentary facies, burial history, tectonics, and other geologic processes. Given sufficient heat, pressure, and time, organic-rich sediments, after deposition, slowly cook and lithify, thus, the organic matter contained within them transforms into kerogen. Categorized by provenance, kerogen is divided into four types: I, II, III, and IV. Additionally, classification based on hydrogen, carbon, and oxygen content reveals distinct implications for the resulting petroleum. Kerogens rich in hydrogen promote the generation of both oil and hydrocarbon gas, whereas those with lower hydrogen content predominantly yield hydrocarbon gas (Nunez-Betelu & Baceta, 1994; Martinelli, 2002; Nieyemer, 2011; Al-Areeq, 2017).

Despite being part of the oil-rich Niger Delta Basin, the study area has not been explored at all for hydrocarbons, probably, due to scarcity of information on its economy viability. The results of this study would, therefore, provide the information required to advance future development in exploration and exploitation of hydrocarbons in this part of the Niger Delta Basin. Samples from shale outcrops in various regions of Ini, Akwa Ibom State, Niger Delta Basin, Nigeria, underwent analysis through Total Organic Carbon (TOC), rock–eval pyrolysis, and vitrinite reflectance measurements. The objective was to characterize the quantity, kerogen type, and thermal maturity of the organic matter within the kerogen, thereby providing substantiation for its potential in hydrocarbon generation.

2.0 Geology/Lithostratigraphy of the study area

The study was conducted in Ini, Akwa Ibom State, Southeastern Nigeria. The study area is located between latitudes 5° 18^{\cdot} - 5° 30 γ and longitudes 7° 17´ - 7° 52´ E, comprising Odoro Ikpe, NnaEnin, Ukwuok, Ndot Ikpe, Oniong Ono, Mbiabong Ikot Udo, Ogu, Ebo, Ikporom, and Obotme communities. The geology of this area has been documented by Okunola & Egbulem, (2015); Udo & Agabi (2019); Asuaiko *et al*.,

(2022) and Ekpo *et al*. (2024). Fig. 1 shows the geology of the study area, extracted from the geology map of Akwa Ibom State, and a cross section from point X to Y.

The lithostratigraphy of the study area was studied at three locations. Coordinates and elevation of each location were established with the Global Positioning System (GPS). Lithostratigraphic units at each location were

described based on observable lithologic properties such as colour, grain size, and mineral composition. Texture and shape of the grains were described using a grain size chart. Each unit thickness was measured with a meter rule or a measuring tape depending on size. The lithostratigraphic logs of the sections as encountered in the field are presented as Figs. 2 - 4.

Fig. 1: Geologic map of the study area showing sample points (Modified after Nigerian Geological Survey Agency, 2012) and a cross section from point X to Y. $L =$ Sample Location

Scale: $1cm = 2m$

Fig. 2: The stratigraphic Section as Encountered in Odoro Ikpe Interpretation: (Modified after Udo et al., 2020).

 $\verb|S| and \verb|stone|$ $\frac{\sigma^{\sigma_{\phi}}}{\sigma_{\phi}}$ Conglomerate ٠, $\frac{1}{2}$ Ĩ, ${\tt Shale}$

Scale: $1cm = 1m$

Fig. 3: The stratigraphic Section as Encountered in Oniong Ono Interpretation: (Modified after Udo et al., 2020).

Sandstone ిం Conglomerate Shale

3.0 Materials and Methods

Geochemical analysis utilized seven shale samples collected from outcrops in Ini, within the Niger Delta Basin, Nigeria. Sample positions and elevations were determined using GPS. Fresh samples were obtained by excavating to a depth of 50 cm with a shovel, and these were then carefully labeled and stored in cellophane bags. After sun-drying, the samples were pulverized

with a mortar and pestle for subsequent geochemical analyses. The determination of total organic carbon (TOC) content involved combusting 100 mg of chemically treated shale samples in the presence of excess oxygen, using the LECO C230 carbon analyzer. The resulting CO2 was quantified by an infrared (IR) detector. The Hawk Rock–Eval instrument was employed for analyzing samples with known TOC, with

parameters commonly used for source rock evaluation. Hydrocarbons were analyzed using a flame ionization detector (FID), while $CO₂$ was measured by a thermal conductivity detector (TCD). Vitrinite reflectance measurements were conducted using a reflected light microscope, specifically the Leica DM 25000 P, equipped with a photometer and a 50x oil immersion objective. 29, 30, and 50 readings were taken from the rock chips prepared from samples 1, 2, and 5 respectively, depending on the abundance of vitrinite in each block. An average of the total number of readings per sample was obtained and recorded as the mean random value (Ro). Vitrinites appear dark gray under normal reflected light and show greenish yellow colour under fluorescence mode as presented by Chaudhuri, 2016. This process followed the Standard Test Method ASTM D7708-14 and Practice D2797. These established methods and procedures have been documented by Tissot & Welte, 1984; Peters, 1986; Peters & Cassa, 1994; Hunt, 1996; Sonel, 2008; Ogala, 2011; Obaje, 2013, Ikwuagwu &Uzoegbu, 2018, Offorbuike *et al*., 2019; Mode *et al*., 2019; and Ndip *et al*., 2019. These analyses were conducted in the laboratory of GeoMark Research Limited in Houston, Texas, U.S.A.

Scale: $2 \text{cm} = 1 \text{m}$

Sandstone $_{\rm State}$

4.0 Results and Discussions

4.1 Organic matter richness

The assessment of organic matter quantity in sedimentary rock commonly involves measuring the total organic carbon (TOC) within the rock. For a rock to be deemed a potential source of petroleum generation, it must contain approximately 1% organic matter, with at least 0.5% TOC. Tissot & Welte, 1984; Conford, 1986; Jarvie, 1991a; Peters & Cassa, 1994; Ehinola *et al*., 2005; Petersen *et al*., 2005; Ndip *et al*., 2019 note that a substantial presence of organic matter is crucial for source rocks to expel oil or gas. Shales from Ini in the Niger Delta Basin, Nigeria, exhibit TOC values ranging from 0.46 wt % to 5.98 wt % with a mean value of 1.65 wt.% (Table 1). According to the classification criteria, TOC and S_1 (the amount of free hydrocarbons in the sample) values-0.0 to 0.5, 0.5 to 1.0, 1.0 to 2.0, 2 to 4, and greater than 4 wt % indicate poor, fair, good, very good, and excellent source rock, respectively (Peters & Cassa, 1994). The

analyzed shale samples designate a source rock with organic carbon content ranging from poor to excellent. Sample 5, with a TOC of 5.98 wt % demonstrates excellent source rock potential, while Sample 4, with a TOC of 0.48 wt % displays poor source rock potential (Table 1). Fig. 5 further illustrates the varied hydrocarbon generation potential of the analyzed samples, ranging from poor to excellent. S_1 values vary between 0.03 to 0.21 mg HC/g rock (Table 1) which signify a poor source rock generative potential (Peters& Cassa, 1994).Peters & Cassa, 1994 proposed that S_2 values between 0.0 and 2.5 mg HC/g rock denote poor source potential. The studied samples with S_2 values ranging between 0.08 and 1.09 mg HC/g rock clearly indicate poor source generative potential. Peters and Cassa, 1994 also recommended that S_2 values from 0.0 to 2.5 mg HC/ g rock denote poor source potential. S_2 data from Table 1 show values that vary between 0.08 and 1.09 mg HC/g rock constituting poor source potential.

Table 1: TOC and Rock Eval Pyrolysis Results and other Derived Parameters of the studied Shale Outcrop Samples from parts of Ini (adapted from Asuaiko et al., 2022)

Sample ID	$\frac{0}{0}$ Carbonate $(wt \, \%)$	TOC (wt $\frac{9}{0}$	$S1$ (mg HC/g)	$S2$ (mg HC/g)	S3(mg) CO2/g)	Tmax($^{\circ}C$	Calc. % R_0 (RE TMAX)	Ш (S2x100) TOC)	OI (S3x100 /TOC	S2/S3 (mg HCl) $mg CO2$)	S1/TOC Norm. Oil	PI $(S1 / (S1 +$ S ₂	GP $S1+S2$
Sample 1	12.03	1.69	0.09	1.00	0.69	398	0.00	59	41		5	0.08	1.09
Sample 2	13.30	1.51	0.08	0.68	0.62	394	0.00	45	41		5	0.11	0.76
Sample 3	11.35	0.62	0.03	0.15	0.46	414	0.29	24	74	θ	5	0.17	0.18
Sample 4	12.49	0.46	0.02	0.08	0.27	427	0.53	17	59	θ	$\overline{4}$	0.20	0.10
Sample 5	18.54	5.98	0.21	1.09	4.41	353	0.00	18	74	$\mathbf{0}$	$\overline{4}$	0.16	1.30
Sample 6	14.94	0.72	0.04	0.08	0.38	413	0.27	11	53	$\overline{0}$	6	0.33	0.12
Sample 7	15.93	0.58	0.03	0.10	0.28	425	0.49	17	48	$\mathbf{0}$	5	0.23	0.13

TOC – Total organic carbon is the amount of organic carbon present in a source rock expressed as weight percent organic carbon per weight of dry rock.

 S_1 – the amount of free hydrocarbons (gas and oil) in the sample generated at the temperature of 300°C.

 S_2 – the amount of hydrocarbon generated from thermal cracking of nonvolatile organic matter at the temperature range of 300° C – 550 $^{\circ}$ C.

 S_3 – the amount of carbon dioxide (CO₂) produced during pyrolysis of kerogen.

Tmax – the temperature at which the maximum release of hydrocarbons from cracking of kerogen occurs during pyrolysis. HI – hydrogen index $(S_2 \times 100/TOC)$, used to characterize the origin of organic matter.

OI – oxygen index $(S_3 x 100/TOC)$, a parameter that correlates the ratio of oxygen to carbon. It is high for polysacharride rich remains of land plants and residual organic matter.

PI - Production Index $[S_1/(S_1 + S_2)]$. PI is used to characterize the evolution level of the organic matter.

GP- Genetic Potential $(S_1 + S_2)$. It is the sum total of hydrocarbon that the source rock can yield.

Fig. 5: A Plot of Depth versus TOC (adapted from Asuaiko et al., 2022)

4.2 Kerogen type

The kerogen concentration in sedimentary rocks varies widely based on source material quantity, preservation, and sedimentation rate. On a global scale, the statistical concentration of organic matter is approximately 1.0%. Rocks with 2% organic matter are considered rich, while those exceeding 5% are deemed very rich (Tissot & Welte, 1984; Nunez-Betelu & Baceta, 1994; Hunt, 1996; Killops & Killops, 2005). Kerogen type classification utilized pyrolysis S2 versus TOC values, hydrogen index (HI), HI versus oxygen index (OI), and hydrocarbon type index (S2/S3) data (Table 1).Studies by Jackson *et al*., 1985; Peters, 1986; Langford & Blanc-Valleron, 1990; Peters & Cassa, 1994; Ehinola *et al*., 2005 underscore the utility of the S2 versus TOC plot for assessing the petroleum generative potential of source rocks. In this investigation, the S2 versus TOC cross-plot (Fig.

6) for shales from Ini in the Niger Delta Basin, Nigeria, indicates predominantly Type III to Type IV kerogen, which suggestsa propensity for dry gas generation(Hunt, 1996). The slopes of lines in Fig. 6, emanating from the origin, are directly proportional to the hydrogen content in the kerogen, with hydrogen content being a critical factor in determining the type of hydrocarbon generated (Hunt, 1996). The HI values for the analyzed shale samples (Table 1) range from 11 to 59 mg HC/g TOC, corresponding to organic matter derived from gas-prone Type III to nongenerative Type IV kerogen as submitted by Peters & Cassa, 1994 that HI values less than 50, $50 - 200$ mg HC/g TOC represent gas to nongenerative Types III and IV kerogen.

The correlation between HI data and oxygen index (OI) data (41-74 mg CO2/g TOC), depicted in Fig. 7, a pseudo van Krevelen diagram

(Espitalie, 1985), indicates kerogen of Type III to Type IV. According to Peter & Cassa 1994, this suggests that the organic matter in the kerogen originates from continental plants.

Fig. 6: S₂ versus TOC cross - plot to identify kerogen type (adapted from Asuaiko *et al.*, 2022)

The hydrocarbon type index (S2/S3) serves as a ratio that reflects the kerogen type, thereby indicating the source rock's potential to expel oil, gas, or both. A hydrocarbon type index falling within the ranges of less than $1, 1 - 5, 5 - 10, 10$ $-$ 15, and greater than 15 mg HC/mg $CO₂$ signifies kerogen Type IV (none-generative potential), Type III (gas generation potential), Type II/III (mixed oil and gas potential), Type II (oil generation potential), and Type I (oil generation potential), respectively(Peters & Cassa, 1994). The S2/S3 data for the analyzed samples range between 0 and 1 mg $HC/mg CO₂$ (Table 1), indicating a predominance of nongenerative to gas-dominant kerogen products (Peters & Cassa, 1994). The confirmation of source rock quality in the analyzed shale samples from Ini, within the Niger Delta Basin, is established through Rock-eval pyrolysis-derived genetic potential ($GP = S1 + S2$). This parameter represents the total amount of petroleum (oil and gas) that could potentially be generated from organic matter in the source rock, given adequate temperature and a sufficient time interval (Tissot & Welte, 1984; Ikwuagwu & Uzoegbu, 2018). The GP value is contingent on the abundance and nature of the organic matter. Source rocks with GP falling below 2, between 2 and 5, between 5 and 10, and exceeding 10 are categorized as having poor, fair, good, and very good generation potential (Hunt, 1996). The analyzed shale samples exhibit a GP ranging from 0.10 to 1.3 mg HC/g of rock, with a mean value of 0.53 mg HC/g of rock, indicating poor generation potential as advanced by Hunt, 1996. These findings align with results obtained by Ehinola et al., 2005; Ogala, 2011; Akaegbobi *et al*., 2017; Ikwuagwu *et al*., 2018; Ndip *et al*., 2019.

Fig. 7: Plot of HI versus OI, determining the type of kerogen present in the source rock (adapted from Asuaiko *et al*., 2022).

4.3 Thermal maturity of organic matter

The alteration of organic matter through heat serves as an indicator of source rock maturity. The concentration of organic matter and the release of hydrocarbons in a source rock depend on the type of organic matter and the extent of thermal changes it has undergone (Tissot & Welte, 1984, Jackson *et al*., 1985). In this investigation, temperature-sensitive parameters such as Rock – eval Tmax $(^{\circ}C)$, Production Index (PI), and measured vitrinite reflectance (%Ro) were employed to evaluate the thermal maturity of organic matter in the source rock. Previous studies by Peters, 1986, Peters & Cassa, 1994; Ehinola et al., 2005; El-Naddy *et al*., 2015; Akaegbobi et al., 2017; Al Matary *et a*l., 2018; Adebowale et al., 2019 have

established the mature oil window to be between $435 - 450^{\circ}$ C. The Tmax values for the examined shale samples, as detailed in Table 1, range from 353^oC to 427^oC, with an average of 403.4^oC, signifying that the organic matter is immature for oil generation. This finding aligns with the results presented in Fig. 8 below.

A cross-plot of kerogen type and maturity, based on Hydrogen Index (HI) and Tmax, was employed to further assess the relationship between the thermal maturity of organic matter and hydrocarbon generation in the analyzed samples, as depicted in Fig. 8 which indicates that the analyzed samples belong to kerogen Type III and are situated within the immature zone. These findings corroborate the results observed in Figs. 6 and 7.

Fig. 8: Plot of HI versus Tmax to indicate source rock thermal maturity

Production Index (PI) values were obtained from the formula $PI = S_1 / (S_1 + S_2)$ as shown in Table 1. The parameter represents the proportion of free hydrocarbons already generated (bitumen) to the amount of hydrocarbon that can be generated (bitumen plus kerogen) (Tissot & Welte, 1984). PI values of less than 0.10, 0.10 to 0.25, 0.25 to 0.40 and > 0.40 indicates that organic matter is in the immature, early mature, peak mature, and late mature stage (Peters & Cassa, 1994). PI values of the analyzed shale samples range from 0.08 to 0.33, with an average value of 0.18. PI values of sample 6 (0.33) , and sample 7 (0.23) are greater than 0.2, indicating a likelihood of migrated oil contents in the shale samples (Peters, 1986). PI values, however, reveal that the stage of kerogen maturation of shales in parts of Ini, Niger Delta Basin is within immature to peak mature windows, indicating immature, none – generative to gas prone source rocks.

To further evaluate the extent of temperature on the kerogen, samples were characterized on a kerogen quality plot of PI against Tmax proposed by Katzs, 1995 (Fig. 9). All the samples fall within the immature zone. It is evident from Fig. 9 that the organic matter in the samples is thermally immature. This is consistent with Peters, 1986; Peters & Cassa, 1994 who proposed that a Tmax value less than 435° C indicates immature organic matter.

Fig. 9: Kerogen conversion and maturity indicators of the studied samples

Vitrinite reflectance measurements on samples 1, 2, and 5 (samples with the highest TOC) yielded mean reflectances of 0.37 %, 0.38% and 0.37%, respectively, indicating that the analyzed shale samples are thermally immature for hydrocarbon generation. This is corroborated by the calculated vitrinite reflectance values for samples 3, 4, 6, and 7 which vary from 0.27 to 0.53 % Ro (Table 1), inferring that samples are in the immature oil generation window. The measured and the calculated vitrinite reflectance values are in agreement with Peters & Cassa, 1994, who proposed that a vitrinite reflectance value of 0.2 to 0.6 indicates thermally immature source rock. Hence the samples are in consonance with the results of Figs. 7 -9. Visual kerogen analysis utilized reflected and fluorescent light observations to assess maceral proportions in volume percent. Photomicrographs of samples 1, 2, and 5 both in reflected and fluorescent lights are presented as Plates 1, 2, and 3. Following the interpretation guidelines proposed by Mastalerz et al., (2018), Plates 1 and 2 reveal rare and small vitrinite of less than 1 volume %, sporadic or scattered inertinite of between 1 - 5 volume %. Liptinite macerals are represented by greenish-yellow

fluorescing liptodetrinite, possibly of both algal and spore origins. Plate 3 reveals that vitrinite is dominant, > 10 volume %, and occurs as relatively large concentrations. Inertinite is sporadic, between 1 - 5 volume %. Liptinite macerals are represented by greenish-yellow fluorescing liptodetrinite, dominantly of pollen and spore origin. Pyrite is rare, less than 1 volume % in Plates 1, 2, and 3. These indicate immature source rocks where the organic matter is mostly unchanged and has not generated hydrocarbon, hence the dark colouration which will tend to absorb light rather than reflect. The maturity level of vitrinite reflectance values is supported by the colour and intensity of the fluorescence of liptinite. The change of fluorescence colours has also been frequently applied as an indicator of thermal maturity (Mao *et al*., 1994). Greenish yellow alginite fluorescent colour (Plate 1) corresponds to vitrinite reflectance of $\langle 0.38\%$ while greenish yellow spore fluorescent colour (Plate 3) corresponds to vitrinite reflectance of 0.25 % (Mao *et al*., 1994), ASTM standards (2011), Mastalerz *et al*., (2018). Both values further confirm that the organic matter in the samples is immature for the generation of hydrocarbons.

Plate 1: Photomicrograph of sample 1 showing vitrinite, pyrite and alginate

5.0 Conclusion

Seven (7) shale outcrop samples from Ini, Niger Delta Basin, Nigeria, were analysed to evaluate the thermal maturity and hydrocarbon potential of the source rock. The results reveal that all but one sample have a TOC above the 0.5 wt. % minimum value for a potential source with values ranging from 0.46 to 5.98 wt. %. Low S_1 values (0.02 - 0.21 mg HC/g rock), and S_2 values $(0.08 - 1.09 \text{ mg } H\text{C/g rock})$ signify poor source rock potential.

Organic matter quality (kerogen type), determined by Rock – eval pyrolysis derived S_2 versus TOC plot, HI data (11 -59 m HC/g TOC), HI versus OI plot, S_2/S_3 , and $S_1 + S_2$ data (0.10 – 1.30 mg HC/g rock), all reveal kerogen Types III to IV. This suggests that the organic matter in the kerogen was derived from continental plants, indicating no generative to gaseous hydrocarbon potential should the thermal maturity process continue. Maturity data deduced from measured vitrinite reflectance $(0.37 - 0.38 %$ Ro), calculated vitrinite (0.27-0.53 %Ro), pyrolysis Tmax $(353-427^{\circ}C)$, and PI $(0.08-0.33)$ values indicate that the analysed shale outcrop samples from Ini, Niger Delta Basin, Nigeria, are thermally immature for hydrocarbon generation. These results are in consonance with similar studies on outcrop samples of the Lower Benue Trough, Gongola and Niger Delta Basins, Calabar Flank, Anambra and Mamfe Basins by Ehinola *et al*., 2005; Abubakar *et al*., 2007; Oguanya *et al*., 2017; Ikwuagwu & Uzoegbu, 2018; Adebowale *et al*., 2019 and Ndip *et al*., 2019.

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