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Research Article

SOURCE ROCK AND THERMAL MATURATION OF CAMPANIAN ENUGU SHALE IN ANAMBRA BASIN, SOUTH EASTERN, NIGERIA

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ABSTRACT
Source rock/ thermal maturation of Campanian Enugu Shale in Anambra Basin, Southeastern Nigeria was assessed by Rock-eval pyrolysis. The total organic carbon (Toc) values range from 1.33 to 4.41wt% (averaging 2.64 wt %). The generation potential (G.P) and Hydrogen Index (HI) values range from 0.06 to 29mg HC/g rock to 7.06 to 128 mg HC/g respectively. These values indicate that the sediments have gas generating potential. The organic matter is dominantly gas prone (mostly type III with few type IV). The SOM values range from 735 to 1931 ppm (averaging 1000 ppm) which indicate moderate to good concentration of organic matter. The degree of thermal maturity recorded from the Production Index (PI) (0.02 to 0.08), calculated vitrinite reflectance of 0.52 to 0.73% Ro and Tmax values between 426 and 438°C (averaging 432°C) indicates that the shale samples are thermally immature to generate petroleum.

Keywords: *Anambra Basin, Enugu Shale, Rock-Eval Pyrolysis, Total Organic Carbon, Source Rock, Thermal Maturation*

INTRODUCTION

The Anambra Basin (figure 1) which is a Cretaceous Basin is located in the Southern part of the regionally extensive northeast-southwest trending Benue Trough. It is a synclinal structure consisting of more than 5,000 ft thick of Upper Cretaceous to Recent sediments, representing the third phase of marine sedimentation in the Benue Trough (Akande and Erdtman, 1998; Ladipo, 1988; Ojo *et al.*, 2009). The basin evolved consequently to the Late Jurassic to Cretaceous Basement fragmentations block faulting, subsidence, rifting and drifting apart of the South American and African plates and therefore representing part of the West African Rift System (Fairhead and Okereke, 1987; Genik, 1992; Ojo *et al.*, 2009). Stratigraphic history of the Anambra Basin shows that the basin comprises of the Campanian to Maastrichtian Enugu/Nkoro/Owelli Formation.

This is succeded by the Maastrichtian, Mamu and Ajali Formations. The sequence is capped by the tertiary Nsukka Formation and Imo Shale (Table 1).

Agagu *et al.*, 1985; Ojo *et al.*, 2009; Petters, 1978; Reijers, 1996). The petroleum geology, biostratigraphy and paleoenvironmental description of Anambra Basin have been carried out by many Authors. Agagu and Ekweozor (1982) shows that the Awgu and Nkporo Shales constitute the main source and seal rocks in the Anambra Basin.

Ekweozor and Gormy (1983) described the Nkporo Shale as an example of a marine source rock composed of Type II/III kerogens with low but consistent contribution from marine organic matter. Unomah and Ekweozor (1993), reported that the organic facies of the Nkporo Shale are provincial with the Calabar Flank having the highest oil potential while those in the Anambra Basin and Afikpo Syncline are gas prone.

Chiaghanam *et al.*, (2012, 2013a,b and 2014a,b,c) used the application of sequence stratigraphy, palynological analysis and lithofacies to describe the hydrocarbon potential of Campanian-Maastrichtian in the Anambra Basin.

Odunze and Obi (2013) used the application of sedimentary and sequence stratigraphy in describing the hydrocarbon potentials of Anambra Basin.

Outcrop samples from different locations within Enugu Shale (along Enugu- Port Harcourt expressway and Enugu- Onitsha expressway) were picked; in other to determine the hydrocarbon richness of the Formation.

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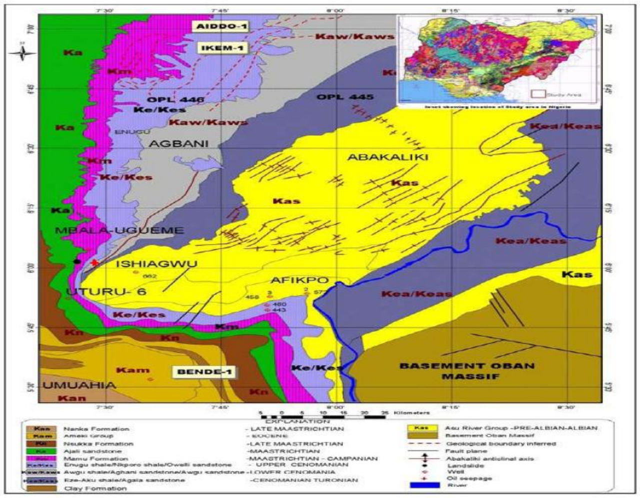

Figure 1: Geologic Map of the Anambra Basin (adopted from Babatunde, 2010)

The objectives of the present study therefore include; (a) To determine the quality of the source rock. (b) To determine and evaluate the thermal and hydrocarbon generation potential of the organic rich sediments of Campanian Enugu Shale.

The deductions from the study will provide information necessary to optimize exploration activities in the Anambra Basin with a view of improving on past investigation in the study area.

Table 1: Lithostratigraphic Framework of Anambra Basin (after Nwajide, 1990)

AGE		ABAKALIKI-ANAMBRA BASIN	AFIKPO BASIN
30 my	Oligocene	Ogwashi-Asaba Formation	Ogwashi-Asaba Formation
54.9 my	Eocene	Ameki/NankaFormation/Nsugbe Sandstone	Ameki Formation
60 my	Paleocene	Imo Formation Nsukka Formation	Imo Formation Nsukka Formation
73 my	Maastrichtian	Ajalli Sandstone Mamu Formation	Ajalli Sandstone Mamu Formation
83 my	Campanian	Nkporo/ Owelli Formation/Enugu Shale	Nkporo Shale/Afikpo Sandstone
87.5 my	Santonian	Non-deposition	
88.5my	Coniacian	Awgu Group (Agbani Sandstone/Awgu Shale)	
93 my	Turonian	Ezeaku Group)	Ezeaku Group (incl Amaseri Sandstone)
100 my	Cenomanian- Albian	Asu River Group	Asu River Group
	Aptian Baremian Hauterivian	Unamed Units	
Precambrian		Basement Complex	

Geology of Enugu Shale

The formation has its type locality at the Enugu Municipality, with an area coverage that stretches north to Ikem-Ihandiagu area, and southwards to Awgu area. It underlies the cross-river plains east of the Enugu Cuesta and largely overlies the Awgu Shales. The formation consists mainly of shales, with two distinguishable sandstone bodies- the Otobi and the Okpaya Sandstones- which are regarded as members of the Formation (Nwajide, 2013). Most of the territory underlain by the formation is low ground except for a few laterite- capped mounds or ridges considered to be erosional resistors left behind at the Cuesta scarp retreated westwards. Relatively consolidated siltstones and/or fine sandstones underlie some of these isolated topographic prominences (Nwajide, 2013).

A mix of native sulphur, gypsum efflorescence, shale fragments, burrows of ichnogenous *Thalassinoides*, growth fault, and Roll-over anticline and normal fault has been observed in the Formation.

The Otobi Sandstone member- Otobi Sandstone member of the Enugu Formation is a relatively small body stretching roughly NNE-SSW from the southern outskirts of Otukpo in Benue State. It overlies the Awgu Shale and laterally interfingers with the shaly facies of the Enugu Formation. It is assumed not to be contiguous with the more extensive Okpaya Sandstone member, which is a stratigraphic unit lying within an area of both the Benue Trough and the Anambra Basin (Nwajide, 2013).

The Okpaya Member- Okpaya Sandstone Member of Enugu Formation underlies a large territory that stretches northwards from Ihandiagu-Ikem area of Enugu State into Benue State, and swings westwards, describing an arch that terminates as an onlap on the basement around north of Idah in Kogi State. Its topographic expression as a low ridge surrounded by the shales of the Enugu Formation facilitates cartographic distinction. Its characteristics are noted at Eke, 35km southwest of Otukpo. It becomes shalier upwards. The whole exposure may be described as a sandy heterolith. Lenticular lamination and bioturbations is intense in places. A mixed tidal flat setting is suggested as the main depositional environment (Nwajide, 2013).

Location and Accessibility-

The study area lies within longitudes 7° 25'E and 7° 35'E and latitudes 6° 00' and 6° 15' and falls within the Enugu Shale of Anambra Basin, see figure 2. The area has good road network and linked up by Enugu-Port-Harcourt and Enugu-Onitsha express-ways.

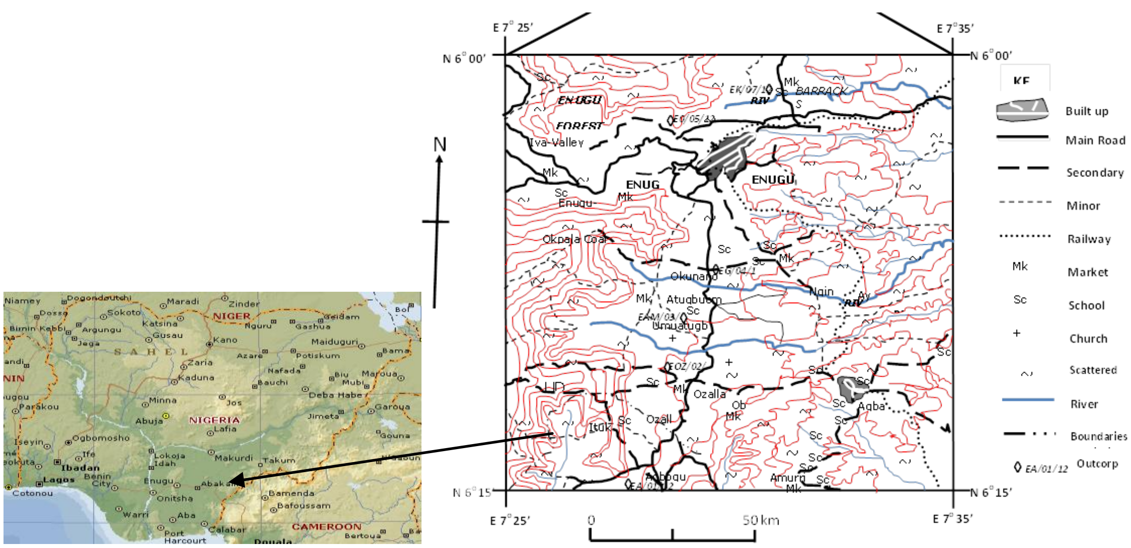

Figure 2: Location Map of the Study Area

MATERIALS AND METHODS

Twenty-seven shale samples recovered from Enugu Shale outcrops along Enugu-Port-Harcourt, Enugu-Onitsha express-ways and Emene River were selected for Total organic content determination, Rock-eval

Pyrolysis and Soluble Organic Matter determination which was aimed towards understanding the hydrocarbon generating potential, maturity and type of Kerogen of Enugu Shale

Total organic content (Toc)- the samples were subjected for preliminary total organic carbon content determination by using Walkley Black wet oxidation method, which involves subjecting 0.5g of each pulverized samples to chromic oxidation using the principles of Walkley and Black (1965). This served as a preliminary step towards carrying out Rock-eval pyrolysis analysis.

Rock-eval Pyrolysis- The samples were subjected to Rock-eval Pyrolysis using the Principles and procedures according to Espitalie *et al.,* (1985) and Stach *et al.,* (1982). The samples were heated in an inert atmosphere to 550°C using a special temperature programmed. The parameters/ results obtained from the analysis includes; (1) *First Peak (S_1)* hydrocarbon generated at the temperature of 300°C, (2) *Second Peak (S_2)* hydrocarbon yield from cracking of kerogen and heavy Kerogen in the rock sample at the temperature of range of 300-550°C. (3) *Third Peak (S_3)* carbondioxide (CO_2) generated during the process of thermal cracking of Kerogen. (4) *Tmax* which is the value of thermal maturity and corresponds to the Rock-eval pyrolysis oven temperature (°C) at maximum S_2 generation (Peters, 1986). (5) *Production or Productivity Index [$PI=S_1/(S_1+S_2)$].* (6) *Hydrogen Index [$HI=(S_2/Toc) \times 100$, mg HC/g Toc].*(7) *Oxygen Index [$OI= (S_3/Toc) \times 100$, $mgCO_2$/g Toc].* (8) *Calculated Vitrinite Reflectance (R_o).*

Determination of Soluble Organic Matter

Soluble Organic matter was extracted by subjecting the sample in standard soxhlet extractor using dichloromethane for 24 hrs. The bitumen obtained were filtered at normal room temperature and weighted in part per million (ppm) (Ojo *et al.,* 2009).

RESULTS AND DISCUSSION

Several workers have demonstrated the usefulness of organic geochemical methods in assessing the generative potential and characteristics of source rocks (Peters and Cassa, 1994; Baskin, 1997; Peters, 1986; Akande *et al.,* 1998; Akande, 2002). In this study, the petroleum potential (quantity), Kerogen type (quality) and level of thermal maturity of the studied samples of the Campanian Enugu Shale were discussed based on Rock-eval pyrolysis data, Total Organic carbon (Toc) and Soluble Organic Matter (SOM).

Organic Matter Richness- Total organic carbon content (Toc) and Rock-eval analysis were performed on 27 shale samples that are presumed to be source rocks. Total Organic carbon in a source rock comprises of three basic components. (a) Organic Carbon in a retained, hydrocarbons as received in the laboratory; (b) Organic carbon that can be converted to hydrocarbons, called convertible carbon (Jervie, 1991) or reactive or labile carbon (Cooles *et al.,* 1986); and (c) a carbonaceous organic residue that will not yield hydrocarbon because of insufficient hydrogen commonly referred to as inert carbon (Cooles *et al.,* 1986; Jarvie, 1991; Ogala, 2011).

The Toc of the shale samples ranges from 1.33 to 4.41wt% with average value of 2.64 wt% (table 2). The average Toc value in the samples (2.64 wt%) indicates a very good organic matter concentration (Herdberg and Moody, 1979; Hunt, 1979; Peters and Cassa, 1994), Fair to Good Kerogen quality.

The quality of the source rock in the studied area is confirmed by the pyrolysis- derived generative potential (G.P.= S_1+S_2). The Rock-eval pyrolysis revealed that the total hydrocarbon generative potential of the samples fluctuates between 0.6 to 7.06 mg HC/g rocks (Table 2) with an average of 2.77 mg HC/g rocks. S_1 measures hydrocarbon shows as the amount of free hydrocarbon that can be volatilized out of the rock without cracking the Kerogen (mg HC/g rock).

S_1 increases at the expense of S_2 with maturity while S_2 measures the hydrocarbon yield from cracking of Kerogen (mg HC/g rock) and heavy hydrocarbon and represents the existing potential of a rock to generate petroleum (Peters and Cassa, 1994).

On the average of 2.77 mg HC/g rock, the Enugu Shale shows pyrolitic yields exceeding minimum value required for hydrocarbon source rock. Based on Tissot and Welt's (1984) classification, the studied area can be regarded as having moderate to fair oil source rock potential.

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Sample No.	Cordinate	TOC (wt. %)	SOM (PPM)	SOM/TOC (mg) (Mg HC/g rock)	S_1 (mg HC/g rock)	S_2 (mg HC/g rock)	S_3	SP (S_1+S_2)	Tmax (^{o}C)	PI	S_2/S_3	Calc Ro	OI	KEROGEN TYPE	HI	PCI
EA/01/01	7° 27' 37" E 6° 15' 13" N	3.18	1931	60.72	0.09	0.85	0.79	0.94	429	0.03	1.07	0.56	19.00	III	60.12	0.78
EA/01/02		2.76	995	36.05	0.03	1.6	0.8	1.63	430	0.02	6.15	0.58	15.10	IV	39.38	1 .35
EA/01/03		3	938	31.26	0.03	1.14	1.11	1.17	426	0.05	1.03	0.51	23.15	III	49	1.17
EA/01/04		3.3	863	26.15	0.1	1.66	0.64	1.76	428	0.07	3.07	0.53	42.50	III	69	1.43
EA/01/05		2.45	1778	72.57	0.07	5.27	0.7	5.34	430	0.04	18.14	0.58	80.19	III	53	1.11
EA/01/06		2.28	1931	84.6	0.12	6.94	0.82	7.06	435	0.03	6.06	0.67	21.13	III	87	1.70
EO/02/01	7° 28' 45.5" E 6° 08' 59.3" N	2.92	1024	35.06	0.1	1.15	1.41	1.25	431	0.08	0.81	0.59	48.39	III	79	1.03
EO/02/02		1.33	872	65.56	0.01	2.07	0.89	2.08	427	0.07	7.13	052	24	IV	29	1.73
EO/02/03		1.99	856	43.015	0.02	1.96	0.82	1.98	430	0.16	2.39	0.58	40.35	IV	30	1.64
EO/02/04		1.9	753	39.631	0.01	1.26	0.49	1.27	433	0.05	2.57	0.64	80.00	III	71	1.05
EO/02/05	7° 30' 49" E 6° 28' 30" N	2.59	956	36.911	0.03	2.01	0.82	2.04	434	0.06	2.45	065	35.20	III	83	1.69
EO/02/06		3.2	578	18.0.62	0.11	3.22	1.2	3.33	429	0.07	5.64	0.56	32.00	IV	34	2.76
EO/02/07		1.8	942	52.33	0.1	2.97	1.24	3.07	435	0.05	12.37	0.67	48.50	III	106	2.55

EO/02/08		1.5	756	50.4	0.07	1.3	1.19	1.37	429	0.05	6.84	0.56	53.2	IV	46	1.13
EG/03/01		1.56	634	40.64	0.1	1.07	0.54	1.17	433	0.06	3.14	0.64	28.98	III	100	0.97
EG/04/01	7° 29' 23" E 6° 20' 56" N	3.38	1,003	29.67	0.16	0.93	0.64	1.09	428	0.07	3.87	0.53	45.28	III	114	0.90
EG/04/02		4.41	1927	43.69	0.19	5.34	1.2	5.53	438	0.06	4.91	0.73	48.25	III	125	2.92
EG/04/03		2.76	980	35.50	0.06	1.43	0.53	1.49	429	0.04	4.33	0.56	52.50	III	60	1.24
EG/04/04		2.94	986	33.53	0.13	2.05	1.25	2.18	430	0.05	8.20	0.58	34.50	III	50	1.81
EG/04/05		2.58	735	28.48	0.21	4.09	1.49	4.3	429	0.07	8.34	0.56	129	III	106	3.57
EK/04/06		2.76	1,415	51.26	0.11	5.21	1.57	5.32	438	0.03	5.63	0.73	40.20	III	69	2.75
EK/04/07	7° 29' 06" E 6° 29' 49" N	2.52	1,602	63.57	0.17	2.74	1.82	2.91	431	0.02	3.34	0.59	39.50	III	125	2.42
EN/05/01		4.41	895	20.29	0.23	4.62	1.7	4.85	435	0.07	2.12	0.67	48.97	III	49	3.19
EN/05/02		1.56	873	55.96	0.13	5.53	1.29	6.66	438	0.04	8.05	0.73	38.90	III	82	1.38
ER/06/01	7° 34' 00" E 6° 28' 33" N	4.23	1476	34.89	0.21	0.39	0.38	0.6	438	0.07	1.02	0.73	24.40	III	128	0.49
ER/06/02		1.8	995	55.27	0.3	2.74	0.96	3.04	433	0.06	3.60	0.64	28.13	IV	47	2.52
ER/06/03		2.22	1052	47.38	0.13	1.3	0.98	1.43	428	0.05	3.42	0.53	25.30	III	60	1.18
	Average	2.64 wt %	1000 ppm	44.16 mg/g	0.11 mg/g	2.62 mg/g	1.01 g	2.77g	432 °C	0.06 g	5.02	0.58	28.60	III	67.61	1.72

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Hydrogen Index (HI) values for the studied samples ranges from 29 to 128 mg HC/g Toc, with an average of 67.61 mg HC/g Toc, which indicates a source rock with gas generating potential and an atomic H/C range of 0.7 to 1.0 (Peters and Cassa, 1994; Tissot and Welte, 1984). The Soluble Organic Matter (SOM) values were obtained to further assess the organic matter concentration of the formation (Idowu *et al.*, 1993; Beaker, 1972). The SOM values range from 735-1931 ppm with average of 1000ppm, this indicates moderate to good concentration of organic matter and therefore fall within the range of adequate source rocks (Unomah and Ekweozor, 1993).

Types of Organic Matter- The type of organic matter in sediments of the studied area was analyzed by Rock-eval Pyrolysis (table 2). Most of the studied shale units from 27 samples assessed by Rock-eval pyrolysis are mainly type III with few types IV Kerogens present. This implies that the studied area contain Kerogens that shows low atomic H/C (<1.0) and high O/C (<= 0.3) and is gas prone. The plots of Rock-eval S_2 versus Toc (Figure 3) describe the hydrocarbon potential of the samples. The plot shows that majority of the samples in the studied area have above minimum value required for quality source rock. The relationship between the Hydrogen Index (HI) versus Oxygen Index (OI) (Figure 4) suggests a Kerogen type III with few type IV organic matters which are predominantly gas prone. Plots of HI versus Tmax (maximum temperature of pyrolysis) (Figure 5) and HI versus calculated $\%R_o$ (Figure 6), also suggest that the organic matter in the samples is predominantly type III with few type IV Kerogens.

Thermal Maturity of Organic Matter- thermal maturity provides an indication of the maximum paleotemperature reached by a source rock (Ogala, 2011). The thermal maturation of Anambra Basin has been done by many authors (Akaegbobi and Schmidt, 1998; Akaegbobi *et al.*, 2000; Ogala, 2011). The degree of thermal maturity of Enugu Shale was assessed using pyrolysis- derived parameters such as calculated $\%R_o$, Production Index, Rock-eval, Tmax and thermal alteration index. The Tmax value represents the temperature at which the largest amount of hydrocarbon is produced in the laboratory when a whole rock sample undergoes a pyrolysis treatment. The Tmax of the studied area ranges from 426°C to 438°C with an average of 432°C which indicates that the studied samples are at stage of thermal maturity that is immature with thermal alteration index (TAI) of 1.5 to 2.6. The calculated vitrinite (R_o) reflectance values range between 0.51 to $0.73\%R_o$ with an average of $0.58\%R_o$, which also suggests that the stage of thermal maturity for oil at the studied area is basically immature. Plot of production Index against Tmax shows that the studied area has not reached the required degree of temperature for intensive hydrocarbon generation and expulsion since the Production Index and Tmax are very low. The Production Index $[PI=S_1/(S_1+S_2)]$ for the studied area range from 0.02 to 0.16 with an average of 0.06 suggest a stage of thermal maturity that is immature.

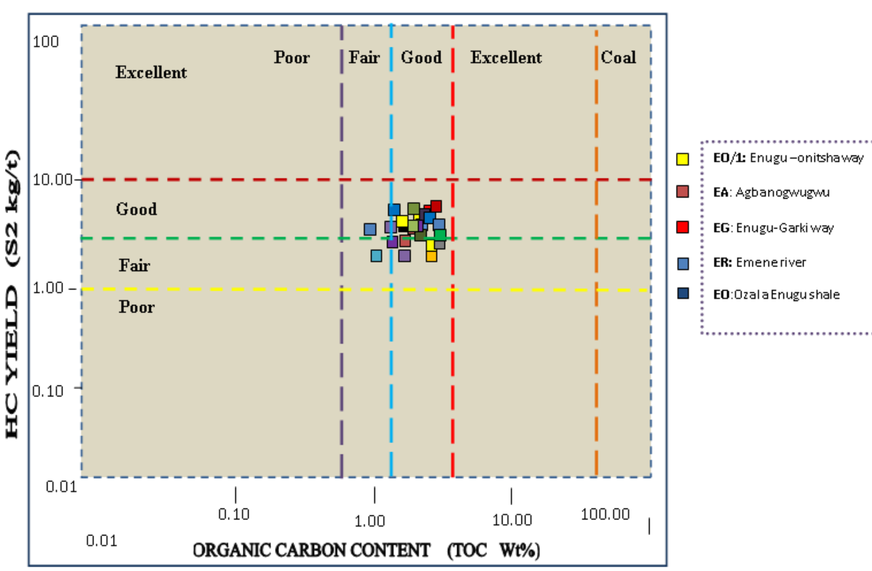

Figure 3: Plot of Hydrocarbon Yield (S_2kg/g) versus Toc (wt %)

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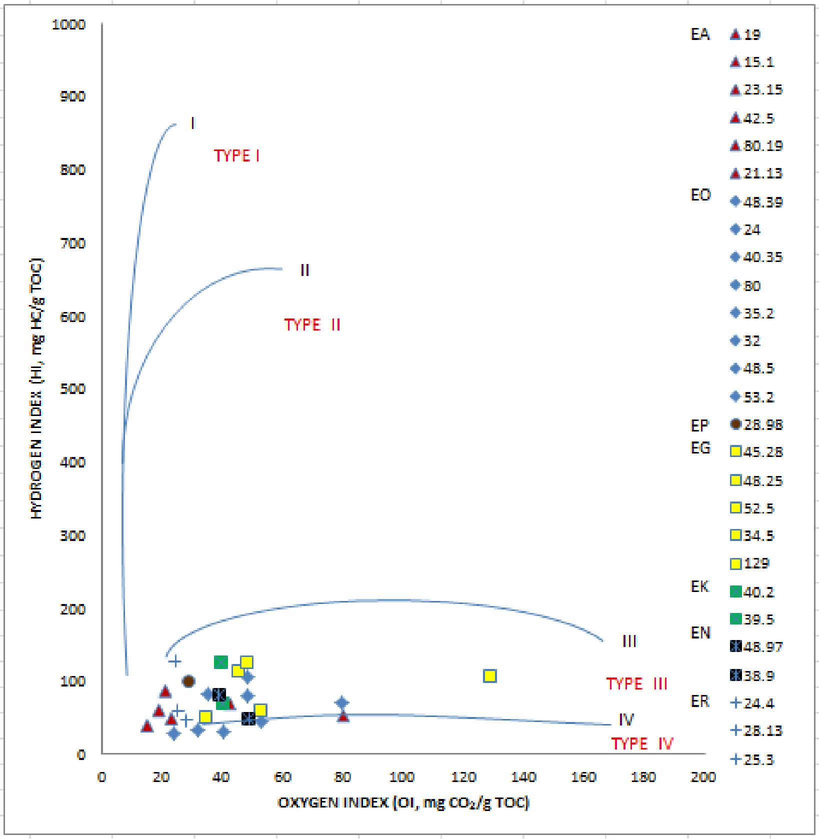

Figure 4: Plot of Hydrogen Index (HI, mg HC/g Toc) versus Oxygen Index (OI, mg CO_2/g Toc)

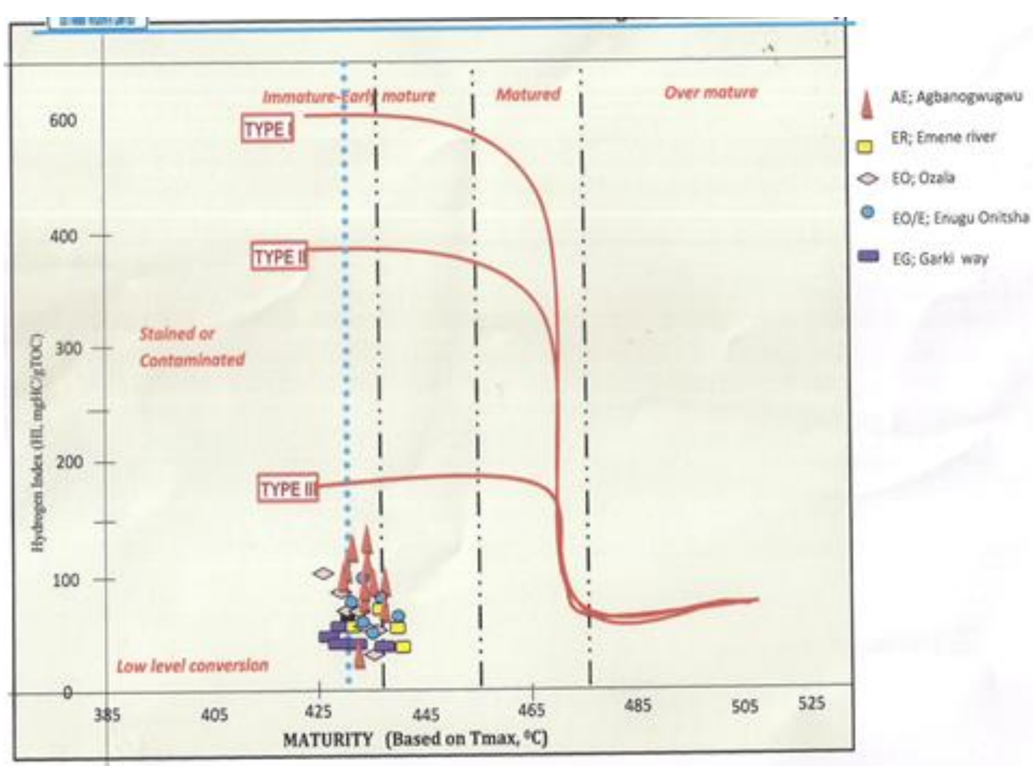

Figure 5: Plot of Hydrogen Index (HI, mg HC/g Toc) versus Tmax

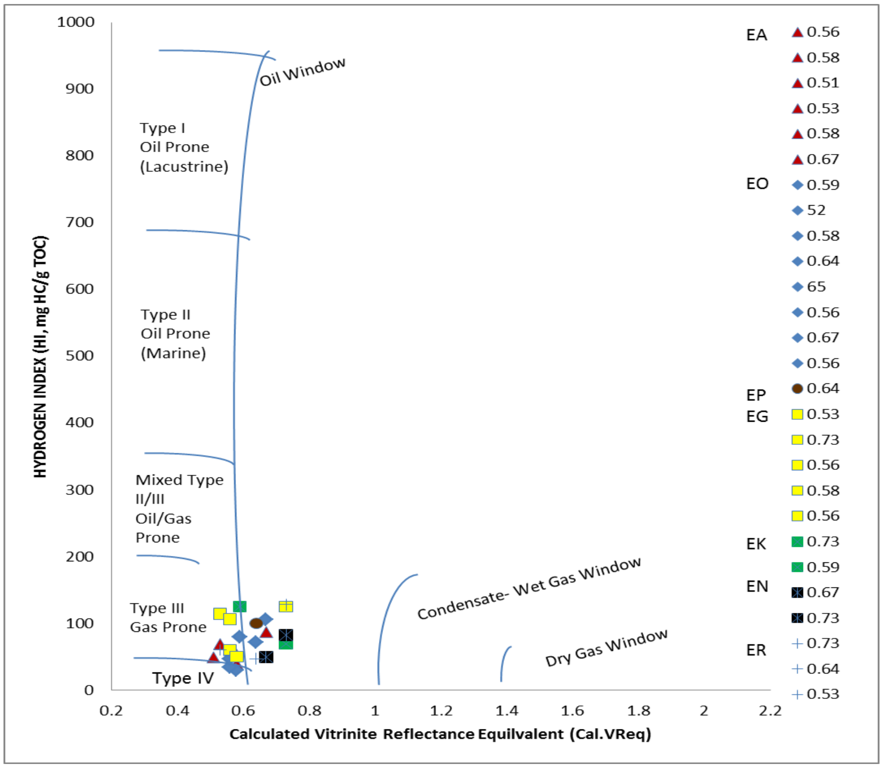

Figure 6: Plot of Hydrogen Index (HI, mg HC/g Toc) versus Calc. Vitrinite Reflectance Equilvalent (Cal. VReq)

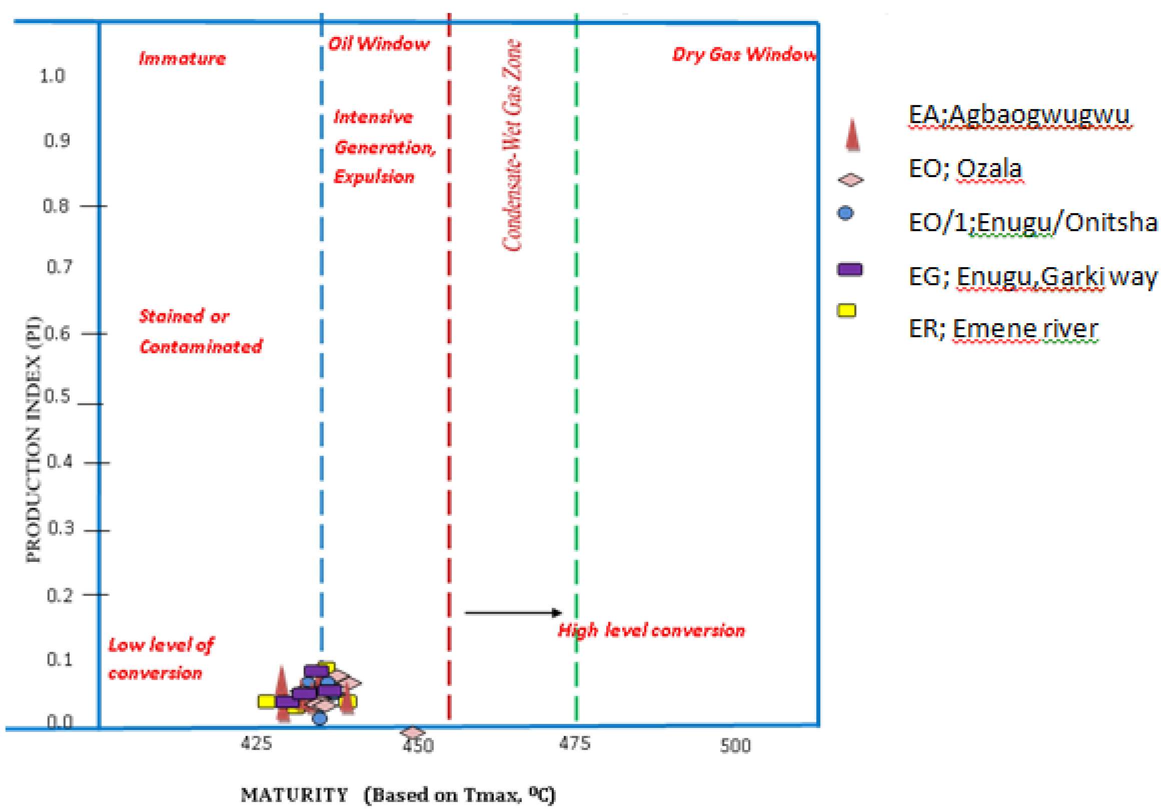

Figure 7: Plot of Production Index (PI) versus Tmax

Conclusion

This study has shown that the shales of Campanian Enugu Shale in the Anambra Basin, South eastern Nigeria has an average total organic carbon (Toc) content of about 2.64 wt%. the Hydrogen Index (HI) and generation potential (G.P) of the shale samples in the study area attained values required for a quality source rock, suggesting that the sediments have gas generating potential and belongs to type III Kerogen with few records of type IV Kerogen. The degree of thermal maturation obtained from the Rock-eval data suggests that the shale sediments are thermally immature to generate petroleum.

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