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Full Length Research Paper

Biomarker evaluation of the oil generative potential of organic matter, in the upper Maastrichtian strata, Anambra Basin, southeastern Nigeria

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Biomarker studies have been carried out on shale samples from the Upper Maastriachtian - Paleocene strata of Nsukka Formation in southern Anambra Basin. The samples were taken on a traverse from Ihube along the Enugu-Port Harcourt expressway through Okigwe to Umuasua near Ovim. Biomarker data from the shales indicate a dominance of intermediate molecular weight n-alkanes (nC_{12}- nC_{33}) and high pristane-phytane (Pr/Ph) ratios. The preponderance of pristane over phytane indicates that the organic matter may have been of humic and toxic to dysaerobic in origin. The n-alkane distribution patterns of the gas chromatograms are non unimodal and display maximum in the range nC_{12}-nC_{33} with no significant humping implying little or no alteration of the organic matter. The carbon preference index (CPI) values range between 1.09 and 1.61.This means that the organic matter is immature and predominantly terrestrial.

Keywords: Biomarker, Kerogene, Maturity, Pristane, Phytane, Provenance

INTRODUCTION

Following the recent federal government signing of a memorandum of understanding (MOU) with foreign investors on the diversification of the gas sector “termed Gas Revolution”, increased exploration and exploitation activities for hydrocarbon in Nigerian inland Basins and off-shore/ultra deep water areas are expected to be intensified. Oil exploration concessions have been acquired in the Anambra Basin by some multinational oil companies operating on a production sharing agreement with the Nigerian government. The Anambra Basin hydrocarbon province appears to hold more gas than liquid petroleum. In the past, the choice had been oil (Agagu *et al.,* 1982; 1985; Akaegbobi and Schmitt,1998; Akande *et al* 1998 & Nwajide,2005) but today, the entire domestic and foreign energy demand scenario is fast changing with increasing demand for liquefied natural gas. Presently, natural gas accounts for over 26% of global energy demand and being a cleaner and environmentally friendly fuel, its demand is expected to rise enormously.

This work aims at evaluating the oil generative potential of the shales from the Upper Maastrichtian-Paleocene Nsukka Formation by assessing the provenance and maturity of the sedimentary organic matter as a preliminary measure to establishing the existence or otherwise of any petroleum potential within the sedimentary successions in the Basin (Figure. 1). The essential elements of any favourable petroleum systems are established mature source rock, regional migration pathways reservoir and seal rocks and favorable trapping and containment structures. The generation of oil and gas from any mature source rock depends on the provenance of the organic matter contained in the sediments. Predominantly terrestrially derived organic matter favours the generation of more gas than oil while a domination of marine organic matter would favour the generation of more oil than gas. This paper approaches the assessment of the provenance and geochemical evaluation of the Nsukka Formation using the distribution and relative proportions of the biomarker compounds extracted from the organic matter in the shales.

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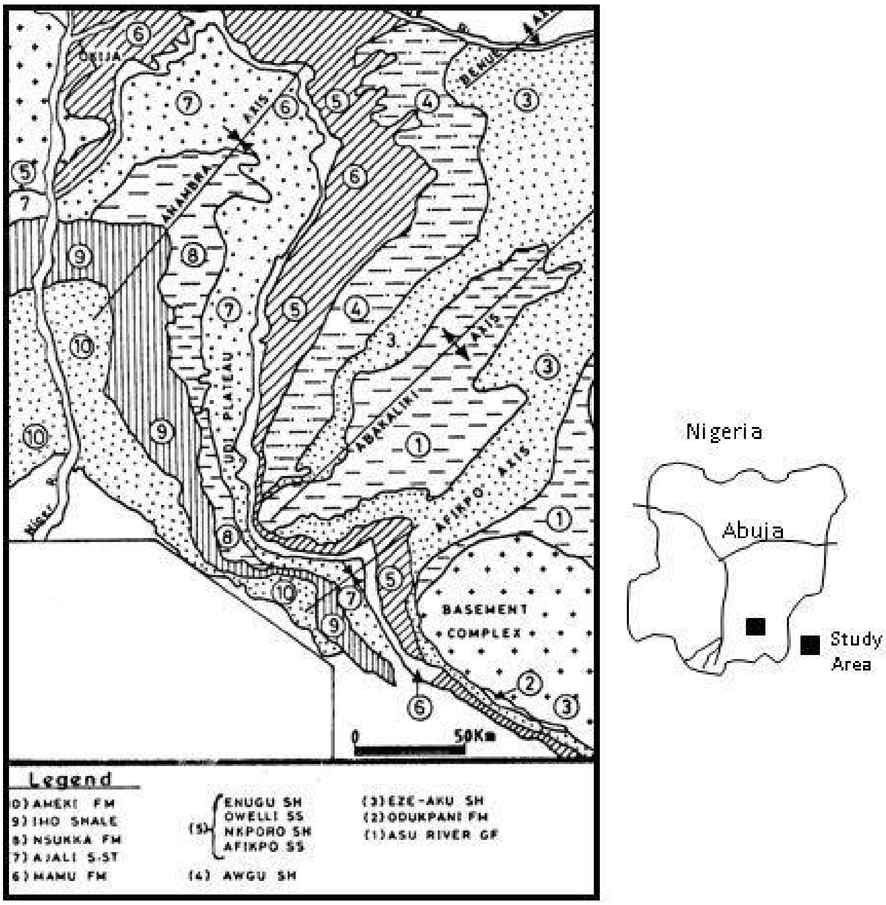

Figure1: Map of Anambra Basin showing the study area (modified from Hoque, 1977).

Regional tectonics and stratigraphy

The oldest sedimentary basin in Nigeria, the Benue - Abakaliki Trough, originated in the early Cretaceous as the failed rift associated with the opening of the south Atlantic (Wright *et al,* 1985). In early Albian – Coniacian periods, two stable areas could be distinguished on either side of the Benue Abakaliki Trough, namely, the Anambra and the Ikpe platforms in the west east of the southern trough respectively. On the southeastern–most part of Trough, the NW-SE trending structure, the Calabar Flank consisting of the Ikang Trough, the Itu High as well as the Eket Platform persisted without significant change right into the Tertiary (Murat, 1972). The Benue - Abakaliki Trough was filled by the Coniacian time. Tectonic movement culminated into the Santonian epierogenic uplift and folding of the Albian – Coniacian sediments into the Abakaliki Anticlinorium along a NE-SW axis. The structural inversion of the trough led to the subsidence of the Anambra Basin and Afikpo Synclines on the west and southeast of the Anticlinorium respectively. These newly subsided sedimentary basins, including the Afikpo Syncline, received sediments from the Campanian to the Paleocene (Figure 2).

The origin and tectonic evolution of the Benue Trough and the Anambra basin have been widely discussed and well reviewed by several authors (Murat,1972; Kogbe, 1976; Petters, 1977; Petters et al., 1982; Hoque and Nwajide, 1984; Benkhelil, 1987; Ojoh, 1992; Reijers and Nwajide, 1998). The sedimentary fill in the Southern Benue Trough consists of three major unconformity-bounded depositional successions: Albian - Cenomanian, Turonian - Coniacian and Campanian – Maastrichtian (Figure 2). Sedimentation commenced with the deposition of Aptian – Albian alluvial fans and marine shales of the Asu River Group, although some pyroclastics also occur (Ojoh, 1992). The Asu River Group consists of sandstones of the Mamfe Formation and shales, limestones and sandstone of the Abakaliki Formation. The Awi Formation and the Mfamosing Limestone in the Calabar Flank are also reported as Albian (Petters et al., 1982). The marine Cenomanian – Turonian Eze - Aku

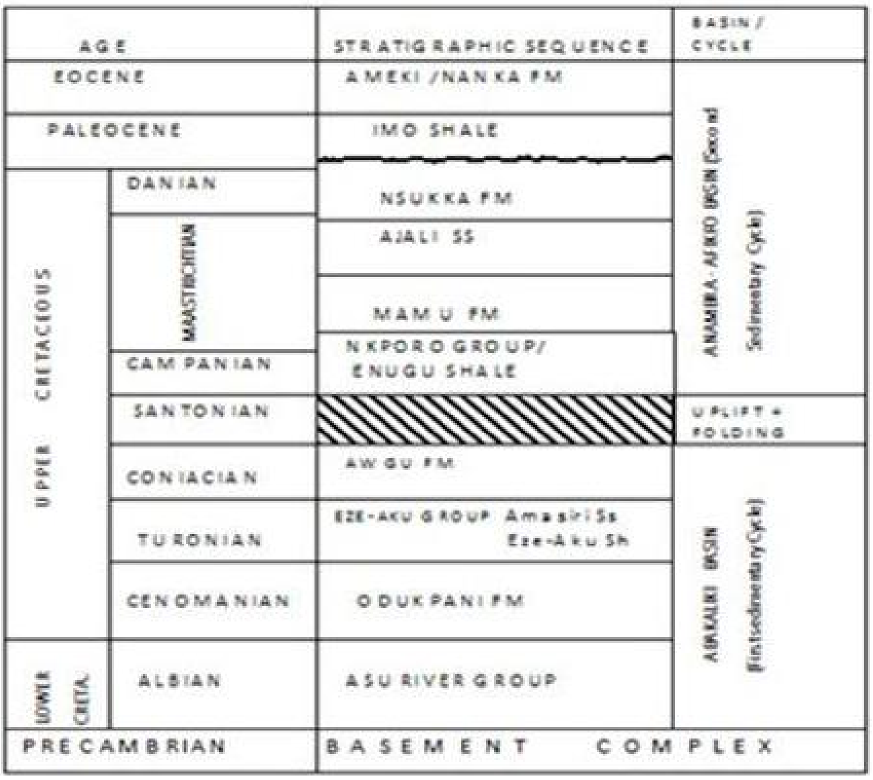

Figure 2: Stratigraphic Framework of Benue Trough and Anambra Basin showing the stratigraphic position of the Nsukka Formation (modified from Reyment, 1965 and Murat, 1972)

Group (black shales, limestones and siltstones) and the inter-fingering regressive sandstones of Agala and Amasiri Formations rest on the Asu River Group. The Coniacian Awgu Shale with the aranaceous facies of the Agbani Sandstone overlies the Eze-Aku Group. The mid-Santonian deformations in the Benue Trough folded these earlier sediments of the trough stage and displaced the major depositional axis west-ward and southeast-wards, creating new depocenters of the Anambra Basin and Afikpo Sycline respectively. Post-deformational sedimentation in the Anambra basin and the Afikpo Syncline commenced with deposition of the Campanian-Maastrichtian marine and parallic shales of the Nkporo Formation and Enugu Formation and their aranaceous facies of the Afikpo and Owelli Sandstones respectively. The Enugu and Nkporo Formations represent the brackish marsh and fossiliferous pro-delta facies of the late Campanian - Early Maastrichtian depositional Nkporo cycle (Reijers and Nwajide, 1998). Deposition of sediments of the Enugu/Nkporo Formations reflects a funnel- shaped shallow marine setting that graded into channeled low energy marshes. These were overlain successively by the Mamu, the Ajali and the Nsukka Formations. The Coal bearing Mamu Formation and the Ajali Sandstone accumulated during this epoch of overall regression of the Nkporo cycle. The Mamu Formation occurs as narrow strip trending north-south from the Calabar Flank, swinging west around the Ankpa Plateau and terminating at Idah near the Niger River. The Ajali Sandstone marks the height of the regression at a time when the coastline was still concave. Two converging littoral drift cells governed the sedimentation and are reflected in the tidal sand waves which are characteristic of the Ajali Sandstone. The Nsukka Formation and Imo Shale mark the onset of another transgression in the Paleocene. The shales contain significant amount of organic matter and may be a potential source for hydrocarbons in the northern part of the Niger delta (Reijers and Nwajide, 1998). Marine shales and sandstones of the Imo Formation and the Ameki Formation were deposited in the Paleocene- Eocene age in the up-dip Niger Delta Basin. They are only locally expected to reach maturity levels for hydrocarbon expulsion (Nwajide, 2005; Obaje et al, 2004). The Eocene Nanka Sand marks the return to regressive conditions with well exposed, sand waves suggesting the

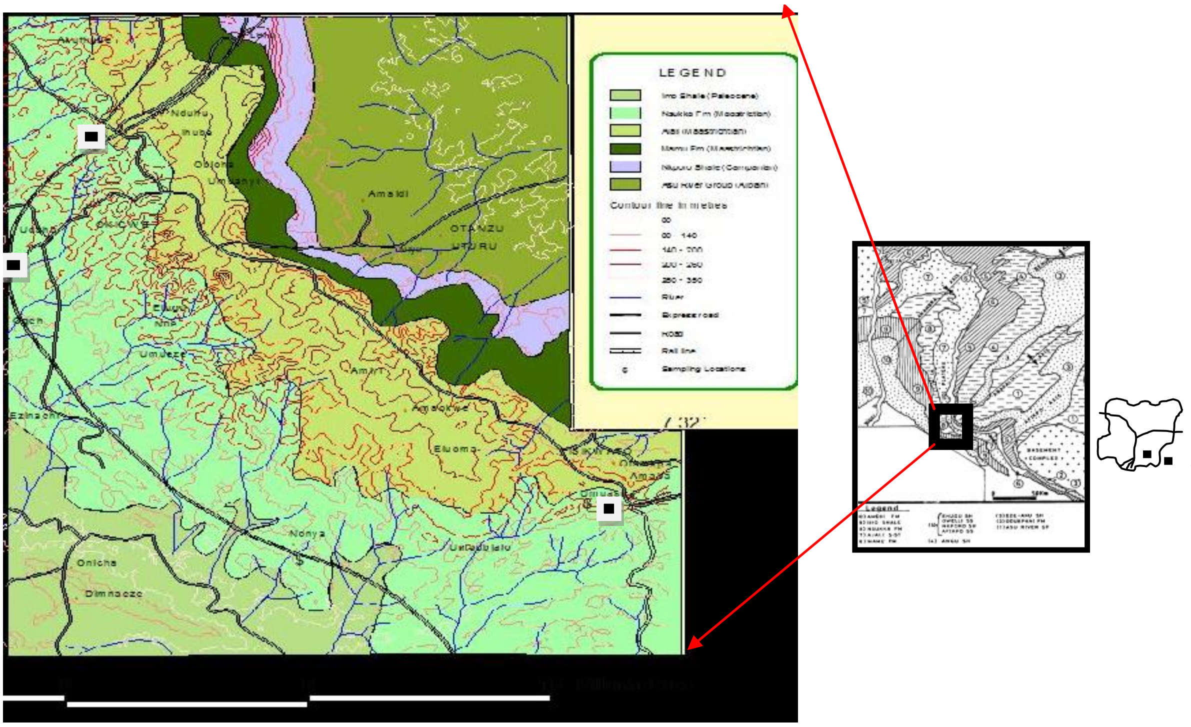

Figure 3: Geologic map of the study area showing the sampling locations.

predominance of flood-tidal currents over weak ebb-reverse currents. The presence of weak ebb-reverse currents is only suggested by the bundling of lamina separated from each other by mud drapes reflecting neap tides. Down dip of the Niger delta, the Akata and the Agbada Formations constitute the equivalents of the Imo and Ameki Formations respectively (Ekweozor, 1982; 2001 & 2004; Ekweozor et al, 1983; 1990). Figure 2 shows the stratigraphic framework of the Benue Trough and the Anambra Basin showing the stratigraphic position of the Nsukka Formation.

METHODS AND MATERIALS

Ten outcrop samples from base of the Nsukka Formation at Okigwe and Umuasua to the top at (Figure. 3) were used for this study. Fresh samples were collected from road cuts, ditches and trenches along the Enugu - Port Harcourt dual carriage express way. After ensuring that there were no impurities or weathering impressions on the samples to be used for the analysis (Dow,1977), 200 mg of pre-cleaned shale samples were crushed and accurately weighed into clean LECO Crucibles. The rock samples were then de-mineralized by hot 10% HCL and afterwards washed repeatedly with distilled water. After drying at 60^{o}C, the crucibles were automatically introduced into the furnace of LECO CS 125 Carbon analyzer for combustion and measurement of the organic carbon contents. Gas chromatography analysis was conducted on a Varian 3400 GC fitted with 45m x 0.25mm fused silica column, coated with a non-polar stationary phase (DB1). Both the injector and detector temperatures were set at 300^{O} C. The oven heating programme was set at 30^{O}C initial isothermal period of 2 minutes; then heated up at the rate of 6^{o}C/min to 300^{O} C, followed by final isothermal period of 13 minutes. The carrier gas was hydrogen set at a flow rate of 2ml/min. Collection and processing of GC data was initially by Atlas software via a chromatographic server. This provided the respective gas chromatograms as well as the corresponding injection reports containing peak heights and distribution of the n-alkanes in the aliphatic fractions.

Lithologic description of outcrops sampled

Three outcrops locations of the Nsukka Formation were logged and sampled for this study (Figure 3). These are the Ihube Section, 1km from the Ihube Junction along the Enugu – Port Harcourt Dual Carriage Expressway (Figures 4a and b); the Okigwe Junction Section and the Umuasua Section, near Ovim in Isiukwuato, Abia State (Figures 4c and d). The Ihube Outcrop comprises of dark

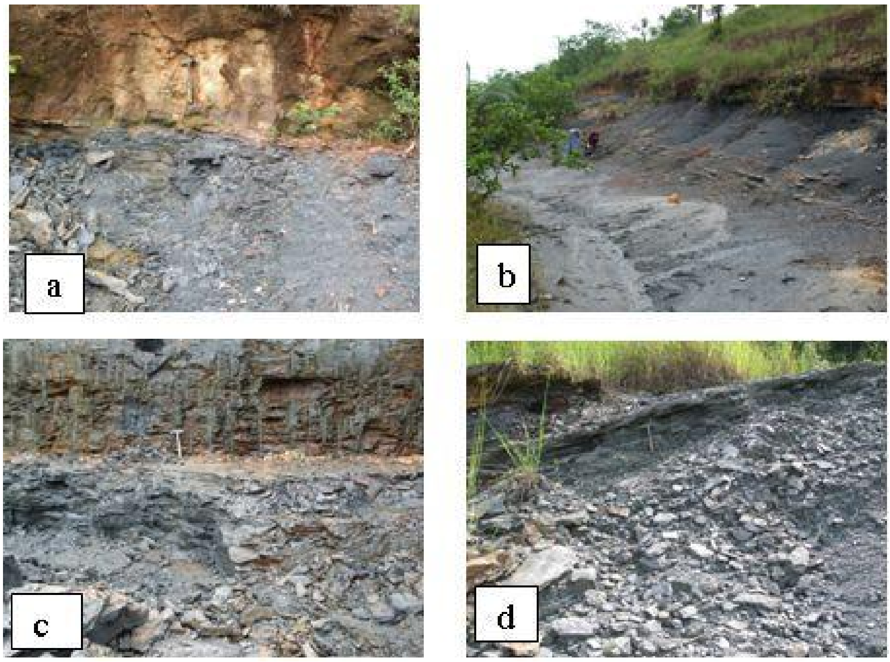

Figure 4: Outcrops of the Nsukka Formation showing well developed dark grey to black shale beds (a and b) at Ihube, along Enugu – Port Harcourt Express way (c and d) at Okigwe Junction and Umuasua respectively.

Table 1. Total Organic Carbon and Rock-Eval Pyrolysis Data Sets

S/N	Client's ID	Lab No.	Sample Type	Toc (wt%)	S_1 (mg/g)	S_1 (mg/g)	S_1 (mg/g)	Tmax (Deg. C)	HI (mg/gToc)	HI (kg/Tonne)	S_1/Toc	PI	VR
1	Okhb 1c	8260	Outcrop	1.38	0.05	0.28	0.38	430	20	0.33	0.04	0.16	0.59
2	Okhb 1D	8261	Outcrop	1.75	0.02	0.33	0.2	426	19	0.35	0.01	0.06	0.59
3	Okhbd 2b	8262	Outcrop	1.08	0.02	0.13	0.41	432	12	0.15	0.02	0.13	0.63
4	Okhbd 2d	8263	Outcrop	1.30	0.03	0.14	0.34	425	11	0.17	0.02	0.18	0.58
5	Umuaz 5d1	8264	Outcrop	1.79	0.12	0.64	0.39	422	36	0.76	0.07	0.16	0.60
6	Umuaz 5e2	8265	Outcrop	1.19	0.01	0.17	0.22	416	14	0.18	0.01	0.06	0.62
7	Umuaz 5e3	8266	Outcrop	1.52	0.00	0.23	0.31	419	15	0.23	0.00	n.a	0.59
8	Oknd 3b	8267	Outcrop	0.80	0.01	0.21	0.44	419	26	0.22	0.01	0.05	0.60
9	Oknd 3c	8268	Outcrop	0.72	0.09	0.12	0.95	430	17	0.21	0.12	0.43	0.61
10	Okumah 4a	8269	Outcrop	0.85	0.02	0.14	0.48	422	16	0.16	0.02	0.13	0.60

NOTE: n.a = not available

grey to black shales overlain by fine to medium grained, bioturbated sandstone. The shale is silty in places, but dominantly muddy and well- laminated. At the Okigwe Junction, a 4 m thick outcrop of dark grey to black shale is overlain by ferruginised, bioturbated sandstone. The shale is hard and well-laminated.

RESULTS AND DISCUSSION

The results of rock-eval pyrolysis of the shale samples revealed that the amount of free hydrocarbons (S_1), and the generated hydrocarbons (S_2) through thermal cracking of non-volatiles are respectively very low with S1 varying from 0.00 to 0.12 mg/g and S_2 from 0.12 to 0.64 mg/g. S_3 and Tmax values are also low varying from 0.2 to 0.95 mg/g, and 419^o C to 432^o C respectively (Table 1). Generally, if S_1 is greater than 1mg/g in any organic rock, it is indicative of hydrocarbon shows and if the Tmax is between 430 ^oC and 440 ^oC, with R_o range of 0.65 - 0.85% in any organic rich rich rock, it is indicative

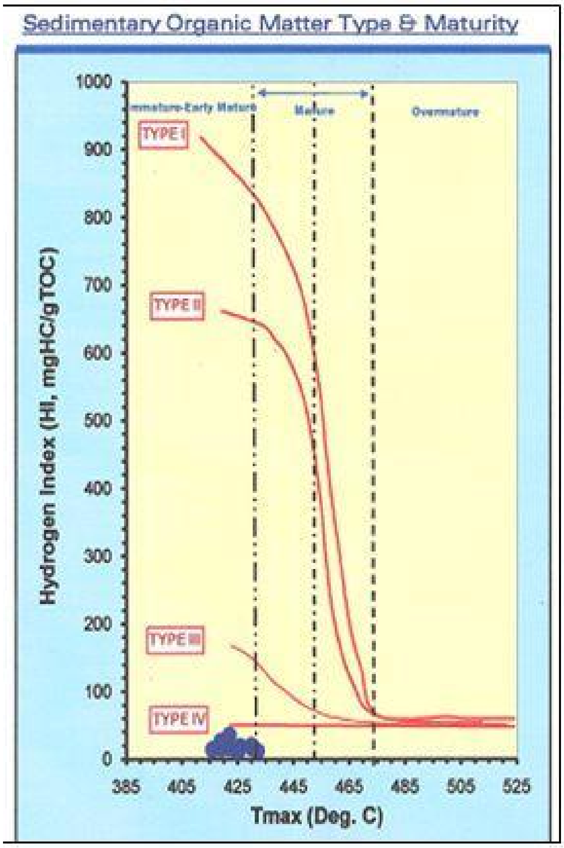

Figure 5: Tmax/Hydrogen index plot showing Type and Thermal maturity of Organic matter.

of threshold hydrocarbon generation (Dembicki *et al,* 1983; Obaje, 2000). But the plot of HI versus Tmax (Figure 5) reveals little or no potential for hydrocarbon generation from these rocks. The HI values with ranges between 11mg/g and 36mg/g TOC (Table 1) confirms the inertness of the kerogen with respect to hydrocarbon generation. This suggests the occurrence of immature to early mature and low quality and quantity of organic matter of the Type IV variety.

The result of gas chromatographic analysis (Figures 6 - 10), reveals marked absence of major n-alkanes especially in the region of n-C_1 to n-C_{11} while n-C_{12} to n-C_{33} alkanes are clearly present without humping or muddling up of the gas chromatogram signatures. This implies that the n-alkanes present in the samples are unaltered and hence not biodegraded. Usually, the humping of the gas chromatogram is associated with unclear and unresolved complex mixture of branched and cyclic hydrocarbons (Wapples, 1980). This probably represents the resistant chemical architecture left after extensive biodegradation or reworked chemical compounds obtained from microbial degradation.

The Pristane (n-C_{17}) and Phytane (n-C_{18}) peaks occur clearly and abundantly in all the samples analyzed. However, the genetical indices (ie isoprenoids distribution) indicate the predominance of Pristane (n-C_{17}) over Phytane (n-C_{18}) which supports humic origin of the organic matter. Also the gas chromatograms reveal that n-alkane distribution pattern in all the samples are non - unimodal and display maximum in the range n-C_{12} – n-C_{33}. The Pristane - Phytane ratios for the samples analyzed varied between 0.30 and 6.10 with a mean ratio of 2.40 (Table 2). This suggests a deltaic to peri-deltaic, oxic depositional environment. Usually, Pr/Ph ratio above 1.00 indicates oxic to dysaerobic environment (Cooper, 1990).

The Carbon Preference Index (CPI) values range between 1.09-1.61 while the vitrinite reflectance (Ro) values are moderate, ranging between 0.58 and 0.63 (Table 2). The visual kerogen analysis showed preponderance of amorphous organic matter (27-53%), subordinate amounts of oxidized vitrinite (8-42%) and primary detrital inertinite (7- 20%) (Table 2). This signifies thermal immaturity to early (threshold) maturity of the kerogen (Demaison *et al*, 1980).

Summary and Conclusion

The extractable organic matter (EOM) obtained from the shale samples of the Nsukka Formation around Okigwe and Umuasua environs were analyzed using a number of analytical procedures in order to assess their hydrocarbon potentials. The presence of immature to probably early/marginally mature low quality and quantity of organic matter of Type IV kerogen was noted. Gas chromatographic analysis revealed marked absence of n-alkanes from n- C_1 to n- C_{11} and the presence of n- C_{12} to n-C_{33} alkanes without any significant humping signatures suggesting that the n- alkanes are unaltered and non-biodegraded. The Pristane (n- C_{17}) and Phytane (n- C_{18}) signature peaks occur clearly and abundantly in all the samples analyzed. However, the genetical indices (i.e. isoprenoid distribution) showed the predominance of Pristane (n-C_{17}) over Phytane (n-C_{18}) which suggests that the organic matter is humic in origin. The n-alkane distribution patterns of the gas chromatograms are non unimodal and display maximum in the range n-C_{12} to n-C_{33}. The mean pristane-phytane ratio of about 2.4 implies that the shales of the Nsukka Formation were deposited under deltaic or peri-deltaic, oxic environmental conditions. Furthermore, the carbon preference index (CPI) between 1.09 and 1.61 from gas chromatograms indicates thermally immature organic matter. The vitrinite reflectance (Ro) values ranges between 0.58 and 0.63 signifying overall thermal immaturity to early (threshold) maturity stages of the kerogens. The result of visual kerogen analysis showed preponderance of amorphous

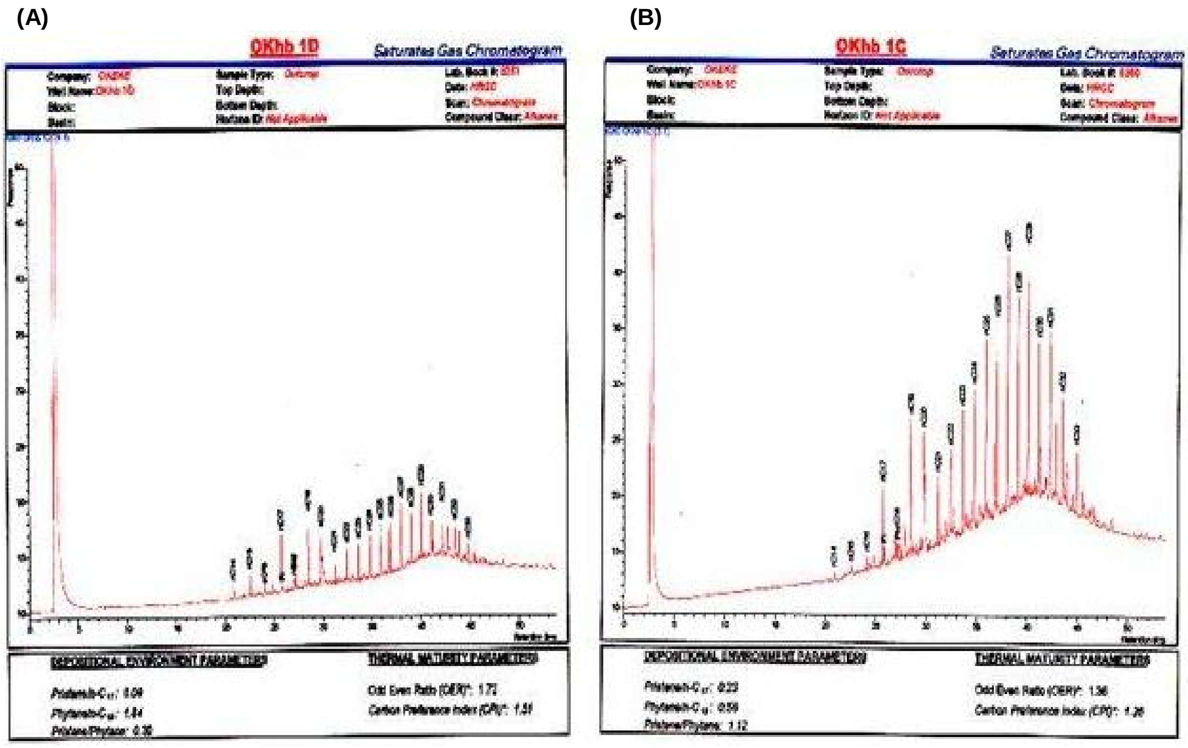

Figure 6(A). Gas chromatogram of sample of sample khb 1D showing depositional Environmental and thermal maturity parameters **(B).** Gas chromatogram of sample of sample Okhb 1c showing depositional environmental and thermal maturity parameters

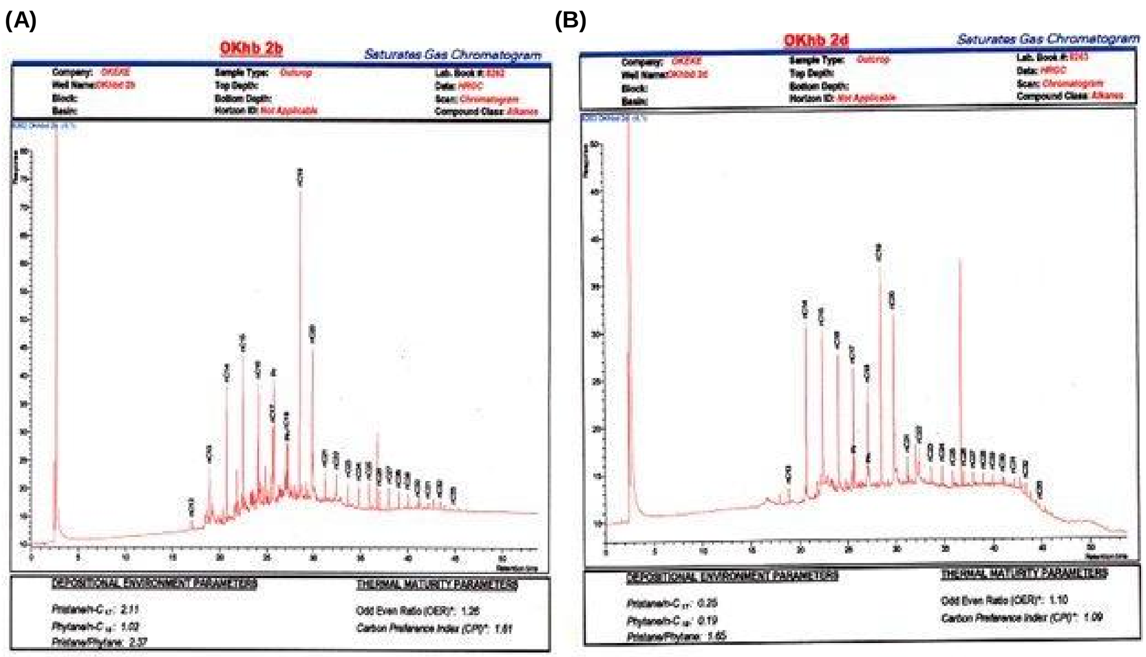

Figure 7**(A).** Gas chromatogram of sample of sample Okhb2b showing depositional environment and maturity parameters **(B).** Gas chromatogram of sample of sample Okh2bd showing depositional environment and maturity parameters

(A) (B)

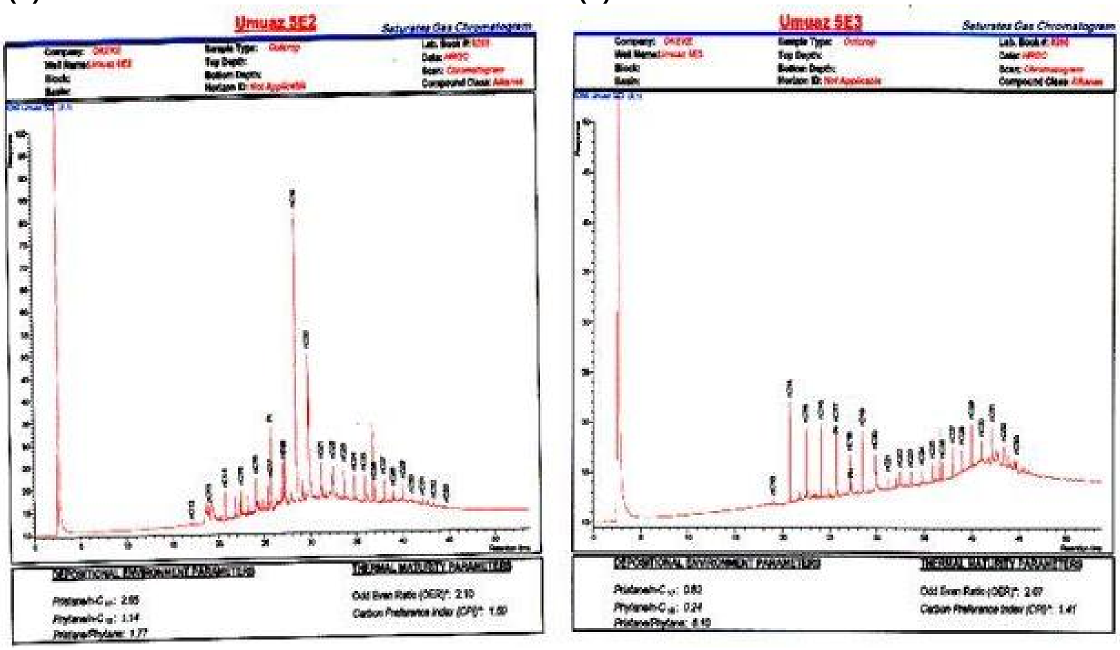

Figure 8**(A).** Gas chromatogram of sample of sample 5E2 showing depositional environment and maturity parameters **(B).** Gas chromatogram of sample of sample 5E3 showing depositional environment and maturity parameters

(A) (B)

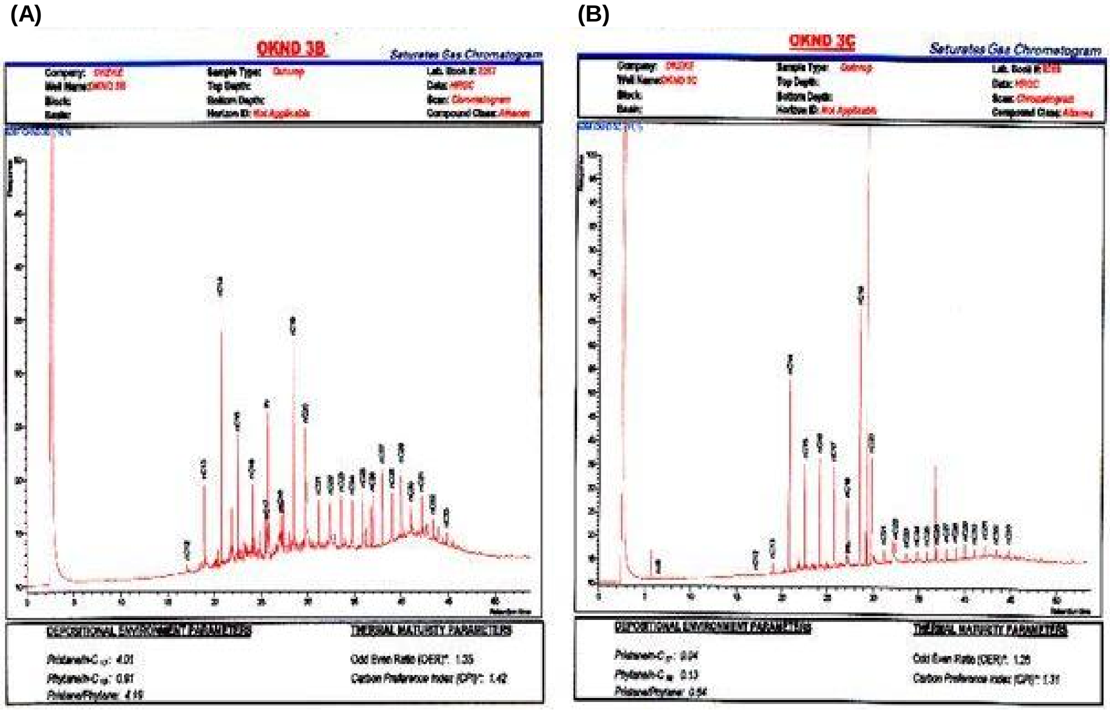

Figure 9**(A).** Gas chromatogram of sample of sample OKND 3B showing depositional environment and maturity parameters **(B).** Gas chromatogram of sample of sample OKND 3C showing depositional environment and maturity parameters

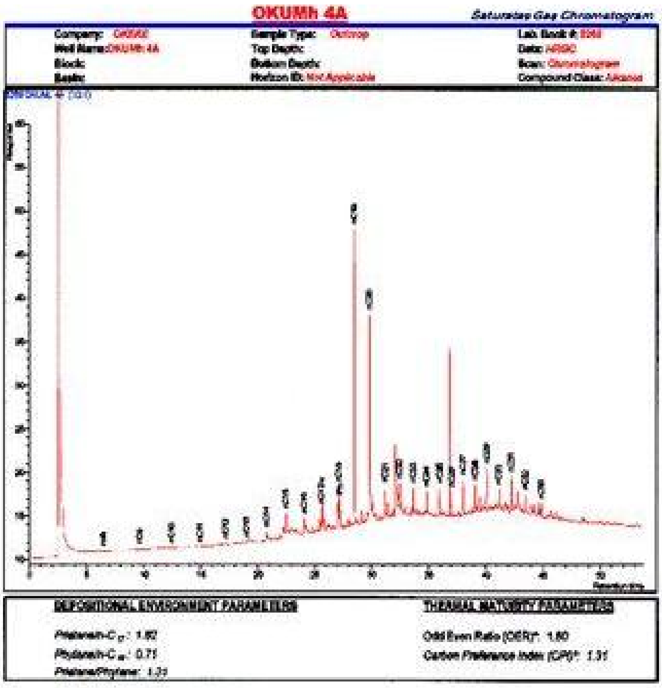

Figure 10. Gas chromatogram of sample of sample OKUMh 4A showing depositional environment and maturity parameters

Table 2: Extractable Organic Matter abundance and molecular parameters in the analyzed samples

S/N	Sample Type	Sample Id	Organic Matter Abundance	Preliminary Molecular Parameters				
			Extractible Organic Matter (mg/g Rock)	Pristine/nC17 (Pr/ C17)	Phytane / nC18 (Ph/ C18)	Pristane/Phytane (Pr/Ph)	Odd-Even Ratio (OER)	CPI
1	Outcrop	8260	29.60	0.23	0.58	1.10	0.23	1.26
2	Outcrop	8261	33.00	0.09	1.84	0.30	1.72	1.51
3	Outcrop	8262	7.70	2.11	1.02	2.37	1.26	1.61
4	Outcrop	8263	13.30	0.25	0.19	1.65	1.10	1.09
5	Outcrop	8264	15.30	0.71	0.32	4.35	1.32	1.25
6	Outcrop	8265	8.60	2.85	1.14	1.77	2.10	1.60
7	Outcrop	8266	31.90	0.82	0.25	6.10	2.07	1.41
8	Outcrop	8267	35.00	4.01	0.91	4.19	1.35	1.40
9	Outcrop	8268	2.00	0.04	0.13	0.54	1.26	1.54
10	Outcrop	8269	5.80	1.82	0.71	1.31	1.80	1.31

organic matter (27-53%), subordinate amounts of oxidized vitrinite (8-42%), primary liptinites (16-33%) and low amounts of detrital inertinite (7-20%). It is therefore concluded that the kerogen in the outcropping shales of the Nsukka Formation may not have attained the requisite maturity to generate hydrocarbon. It is however important to note that the sub-cropping Akata Shales of the down-dip Niger Delta unconformably overlying the Nsukka Formation is the main source rock of the prolific Niger Delta Basin.

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