

Geochemical effects of petroleum generation potential from basal campanian source rocks in the Anambra Basin, S. E. Nigeria

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ABSTRACT

Shale from basal Campanian strata of the Anambra Basin has been characterised by petrological and geochemical techniques. The aims of this study were to assess the quality of its organic matter, evaluate its thermal evolution and highlight its potential as a source rock. The total organic carbon (TOC) (2.55wt%) of the shale constitutes that of a good source rock with gas-prone kerogen indicated by Rock-Eval S2/S3 (2.19). The high oxygen index (OI) (19.09 mgCO₂g⁻¹TOC) and pr/ph (6.50) suggest deposition in a shallow marine environment. The n-alkane distribution dominated by n-C₂₄ – n-C₃₁ and infrared spectrum dominated by aliphatic and aromatic functional groups are considered to be indicators of Type III kerogen. The CPI (1.67), T_{max} (430°C), C₃₁(S/S+R) ratio (0.76) and R_o (0.56%) all indicate the onset maturity of this rock. This thermal evolution is thought to account for its current hydrogen index (41.20 mgHCg⁻¹TOC). Generated petroleum may not have reached the threshold for hydrocarbon expulsion but a review of petroleum system elements in the basin will stimulate high prospects in the Anambra basin.

Key words: Campanian, shale, organic matter, kerogen type, maturity.

INTRODUCTION

Petroleum source rocks are the primary component of the petroleum system concept introduced by Magoon and Dow (1994). They constitute the precursors of petroleum which, under favourable conditions, may subsequently migrate to reservoirs and be sealed to form accumulation. Nigeria's current national petroleum reserves asset (proven), put at 32 billion bbl of oil and about 170 trillion standard ft³ of gas [Nexant, 2003], derives solely from the Niger Delta onshore and offshore. Some exploration campaigns have been undertaken in the inland basins with the aim of expanding the national exploration and production base and thereby add to the proven reserves asset [Obaje *et al.*, 2004]. The inland basins of Nigeria comprise the Anambra basin. The Anambra Basin is a big intra-continental basin that forms an arm of the lower Benue Trough [Obaje *et al.*, 2004] with its NE-SW trending towards the Niger Delta (Fig. 1). Petroleum exploration in the Anambra basin was triggered by the occurrence of surface seeps and dates back to the early 1930s [Kulke, 1995]. However, due to the Santonian inversion and the predominance of terrestrial sediments, as well as discoveries in the prolific Niger Delta in the south [Ekweozor and Gormly, 1983], the petroleum potential of this area has been under exploration and exploitation.

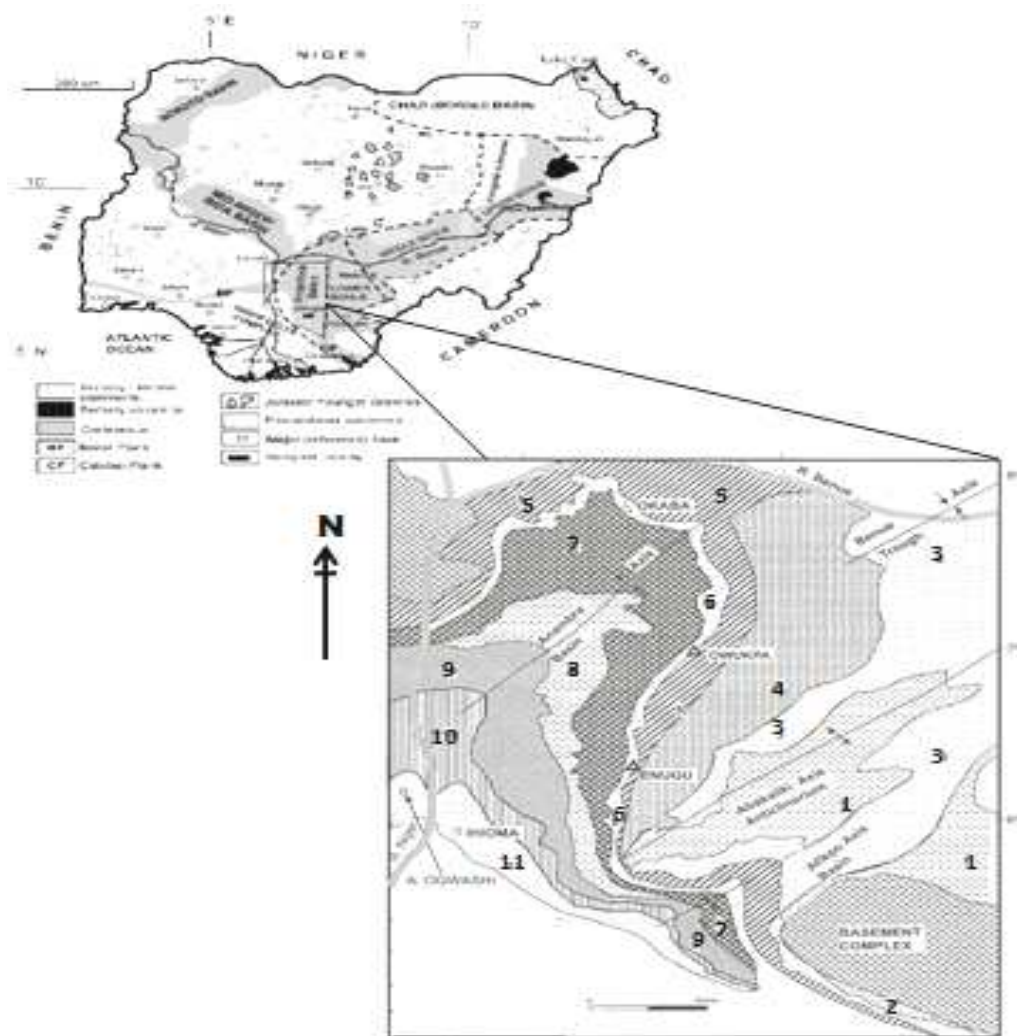


Fig. 1: Generalised geological map of Nigeria showing the location of the Anambra basin and sedimentary basins. Numbers indicate Cretaceous and Tertiary formations shown as follows; 1. Asu River Group; 2. Odikpani Formation; 3. Eze-Aku Shale; 4. Awgu Shale; 5. Enugu/Nkporo Shale; 6. Mamu Formation; 7. Ajali Sandstone; 8. Nsukka Formation; 9. Imo Shale; 10. Ameki Formation and 11. Ogwashi-Asaba Formation [modified from Obaje *et al.*, 2004 and Akande *et al.*, 2007]

Consequently on the exploration campaign, many wells have been drilled especially in the northeast to increase the national reserves but unfortunately most of these wells were reportedly dry. With this development, it has become necessary to adequately evaluate and characterize the petroleum source rocks in this basin by using well established geochemical techniques. At the core of any petroleum system is a good-quality source rock [total organic carbon (TOC) > 0.5%, hydrogen index (HI) > 150 mgHCg⁻¹TOC, liptinite content > 15%, $T_{max} \geq 430^{\circ}\text{C}$, and R_o 0.5-1.2%, biomarker validation]. However, other petroleum system elements must also include, apart from established source rocks, reservoir and seal lithologies, establishable trapping mechanisms, and favourable regional migration pathways [Obaje *et al.*, 2004]. The aims of this study were to characterize a shale sample from the basin using modern techniques of petroleum geochemistry in order to: (i) assess in detail the quality of its organic matter; (ii) evaluate its thermal evolution, and (iii) highlight its potential as a source. The results of this study may stimulate further interest in petroleum exploration and exploitation in the Anambra basin

MATERIALS AND METHODS

The outcrop samples were obtained from the Nkporo shale at Leru 72 km south of Enugu along Enugu – PortHarcourt express road and the Enugu shale at Enugu near the Onitsha-Road Flyover. Care was taking to avoid weathered portions of the outcrop and to obtain material sufficient for various geochemical analyses. The samples were hard, thickly laminated but not fissile, with texture indicative of low permeability. This macro-structure

suggests minimum risk of organic matter oxidation. The samples are attributed to the Campanian-Maastrichtian marine and paralic siliciclastics strata which are overlain by the coal measures of the Mamu Formation (Fig. 2). The fluviodeltaic sandstones of the Ajali and Owelli Formations lie on the Mamu Formation. In the Paleocene, the marine shales of the Imo, and the continental/delta plain shale, mudstone, siltstones and sandstones of the Nsukka Formations were deposited, overlain by the tidal Nanka Sandstone of Eocene age which followed by lignitic Miocene-Oligocene Ogwashi-Asaba Formation.

AGE	SEDIMENTARY SEQUENCE	LITHOLOGY	DESCRIPTION	DEPOSITIONAL ENVIRONMENT	REMARKS		
					Coal Rank	ANKPA SUB-BASIN	ONITSHA SUB-BASIN
MIOCENE	OGWASHI-ASABA FM.		Lignites, peats, Intercalations of Sandstones & shales	Estuarine (off shore bars; Intertidal flats)	Liginites		REGRESSION
OLIGOCENE							
EOCENE	AMEKE / NANKA FM. / SAND		Clays, shales, Sandstones & beds of grits	Subtidal, intertidal flats, shallow marine	Unconformity		(Continued Transgression Due to geoidal Sea level rise)
PALEOCENE	IMO SHALE		Clays, shales & siltstones	Marine			
MAASTRICHTIAN	NSUKKA FM.		Clays, shales, thin sandstones & coal seams	? Estuarine	Sub-bituminous		TRANSGRESSION (Geoidal sea level Rise plus crustal Movement)
	AJALI SST.		Coarse sandstones, Lenticular shales, beds of grits & Pebbles.	Subtidal, shallow marine			
	MAMU FM.		Clays, shales, carbonaceous shale, sandy shale & coal seams	Estuarine/ off-shore bars/ tidal flats/ chemier ridges			
CAMPANIAN	ENUGU/ NKPORO SHALE		Clays & shales	Marine	3 rd Marine cycle	Unconformity	
CONIACIAN-SANTONIAN	AWGU SHALE		Clays & shales	Marine	2 nd Marine cycle		
TURONIAN	EZEAKU SHALE					Unconformity	
CENOMANIAN	ODUKPANI FM.				1 st Marine cycle		
ALBIAN	ASU RIVER GP.					Unconformity	
L. PALEOZOIC	B A S E M E N T C O M P L E X						

Fig. 2: The stratigraphy and environment of deposition of sediments in the Anambra Basin southeastern Nigeria

In the laboratory, the samples were reshaped using a rotating steel cutter to eliminate surface that could be affected by alteration. Chips were cut from the samples and dried in an oven at 105°C for 24 hours. Chips cut perpendicular to bedding were embedded in epoxy and polished following the procedures of Taylor *et al.* (1998) to yield polished blocks for reflectance and fluorescence studies using scan electronic microscope. Another portion of the dried sample was pulverized in a rotating disc mill to yield about 50 g of sample for analytical geochemistry. The total organic carbon (TOC) and inorganic carbon (TIC) contents were determined using Leco CS 200 carbon analyzer by combustion of 100 mg of sample up to 1600°C, with a thermal gradient of 160°C min⁻¹; the resulting CO₂ was quantified by an Infrared detector.

The sample with known TOC was analyzed using a Rock-Eval 6, yielding parameters commonly used in source rock characterization, flame ionization detection (FID) for hydrocarbons thermal conductivity detection (TCD) for CO₂. One milligram of bulk powder sample was added to 200 mg of KBr and the mixture homogenized using a pestle in an agate mortar. Pressing the mixture using a load of 10 t yielded a pellet for Fourier Transform Infrared (FT-IR) Spectroscopy using a Nicolet Bench 505P Spectrometer, with sample absorbance monitored using 256 scans with resolution of 4 cm⁻¹ from a wave-number of 4000 – 400 cm⁻¹. About 10 g of the sample was subjected to sohxlett extraction using a solvent mixture of acetone, chloroform and methanol (47: 30: 23 v/v) at 60°C for 24 hours to extract the soluble organic matter. The extract was concentrated by evaporation to dryness using a rotating vapour evaporator at 250 mb. The extract was transferred to an 8 ml vial using the same solvent mixture and allowed to evaporate to dryness in a vented hood. The dried extract was fractionated by silica gel column chromatography with a column prepared using 2 g of baker silica gel calcined at 200°C for 24 hours to yield six fractions ranging from saturate to polar.

The saturate fraction was subjected to urea adduction to separate isoprenoids from *n*-alkanes and subjected to gas chromatography-mass spectrometry (GC-MS) using a CE 5980 GC coupled to an HP Finnigan 8222 MS held at

80°C for three minutes and raised to 310°C at 3°C min⁻¹ and held isothermally for 10 minutes in order to assess some molecular parameters used in source rock characterization.

RESULTS AND DISCUSSION

Table 1 shows the results of 15 bulk and molecular geochemical parameters used in source rock quality and maturity evaluation. The shale is low in carbonate and its organic matter content within the threshold for petroleum source rocks.

Table 1: Bulk and molecular organic geochemical parameters for shale from the Anambra Basin

Parameter	Result
TOC- Total Organic Carbon (wt%)	2.55
S1 – Free bitumen determined by thermovaporization at 300°C (mgHCg ⁻¹ rock)	0.05
S2 – Amount of kerogen that may be converted to petroleum (mgHCg ⁻¹ rock)	1.10
S3 - Amount of carbon dioxide from carboxyl and carbonyl groups in kerogen (mgCO ₂ /g rock)	0.50
S2/S3- To determine the quality of organic matter	2.19
T _{max} – Temperature at which maximum decomposition of kerogen occurs (°C)	430.00
HI – S2/TOC (mgHCg ⁻¹ TOC)	41.20
OI – S3/TOC (mgHCg ⁻¹ TOC)	19.09
PI – Production Index (S1/(S1+S2))	0.06
R _o – Random vitrinite reflectance (%)	0.56
Pr/ph – pristane/phytane ratio	6.50
CPI – Carbon Preference Index, $\Sigma(^n\text{C}_{23}\text{-C}_{31} + ^n\text{C}_{25}\text{-C}_{33}) / (2 \times ^n\text{C}_{24}\text{-C}_{32})$	1.67
Pr/n-C ₁₇ – pristane/n-heptadecane ratio	6.70
Ph/n-C ₁₈ – phytane/n-octadecane ratio	0.77
C ₃₁ (S/S+R) – homohopane ratio	0.76
GI – Gammacerane index, (gammacerane/17 α -hopane) x 100	9.00

Organic matter quality

The TOC is a primary parameter in source rock appraisal, with a threshold of 0.5-1 wt% at the immature stage for potential source rocks [Tissot and Welte, 1984; Bordenave *et al.*, 1993; Hunt, 1996]. The value of 2.55 wt% of the shale studied exceeds this threshold (Table 1). High TOC of 4.45 wt% was obtained in Mamfe basin and this value exceeds the threshold for oil generation [Eseme *et al.*, 2006]. However, high TOC is not a sufficient condition for oil generation. Coals usually have high TOCs that exceed 50 wt% but do not generate oil except when rich in liptinite, indicating the relevance of maceral composition. In contrast, deltaic sediments may have TOCs below 1 wt% but generate commercial accumulations of petroleum due to deposition of large volumes of sediments, as seen in the Niger Delta. High TOC content in shales indicates favorable conditions for preservation of organic matter produced during deposition. This may related to the redox condition, with high oxygen favoring organic matter oxidation, but also amount of organic matter produced. The high oxygen index of 19.09 mgCO₂ g⁻¹TOC suggests high contribution from terrestrial organic matter poor in hydroxyl groups [Tissot and Welte, 1984] and that the depositional environment was oxic.

The infrared spectrum (Fig. 3) shows a broad band from 1200-900 cm⁻¹, with a peak at 1030 cm⁻¹ attributed to silicate minerals. Principal functional groups related to organic matter include the aliphatics (2920-2850 cm⁻¹) and aromatics (1700-1600 cm⁻¹, 850-700 cm⁻¹). The response at 3500-3400 cm⁻¹, attributed the hydroxyl group, is also influenced by clay minerals and cannot be considered as wholly from organic matter.

The principal application of infra-red spectroscopy is to evaluate the relative importance in organic matter of C=O and C-C functional groups [Tissot and Welte, 1984]. Infrared spectrum shown in Fig. 3 is dominated by the prominent peak from 1200 cm⁻¹ to 900 cm⁻¹, consisting of the response of silicate minerals in the rock. Prominent functional groups related to organic matter include the hydroxyl groups at 3500-3400 cm⁻¹, the aliphatic at 2950-2850 cm⁻¹ and 1450-1350 cm⁻¹, as well as the aromatics at 1700-1600 cm⁻¹ and 850-700 cm⁻¹. The C=O groups attributed to the spectral region of 1710-1700 cm⁻¹ [Mongenet *et al.*, 1999] are absent in Figure 3, but some, attributed to 1400-1040 cm⁻¹ [Tissot and Welte, 1984], may be masked by the broad band attributed silicates. Both the fact that the C=O groups have a high molecular absorption coefficient that is well resolved by FT-IR, and the low intensity of the 1710-1700 cm⁻¹ band relative to other functional groups with lower molecular absorption coefficient, indicate the presence of few carboxyl and carbonyl groups. Aliphatic and aromatic bands dominate the spectrum, with low C=O and OH functionally in agreement with the high OI of 19.09 mgCO₂ g⁻¹TOC (Table 1).

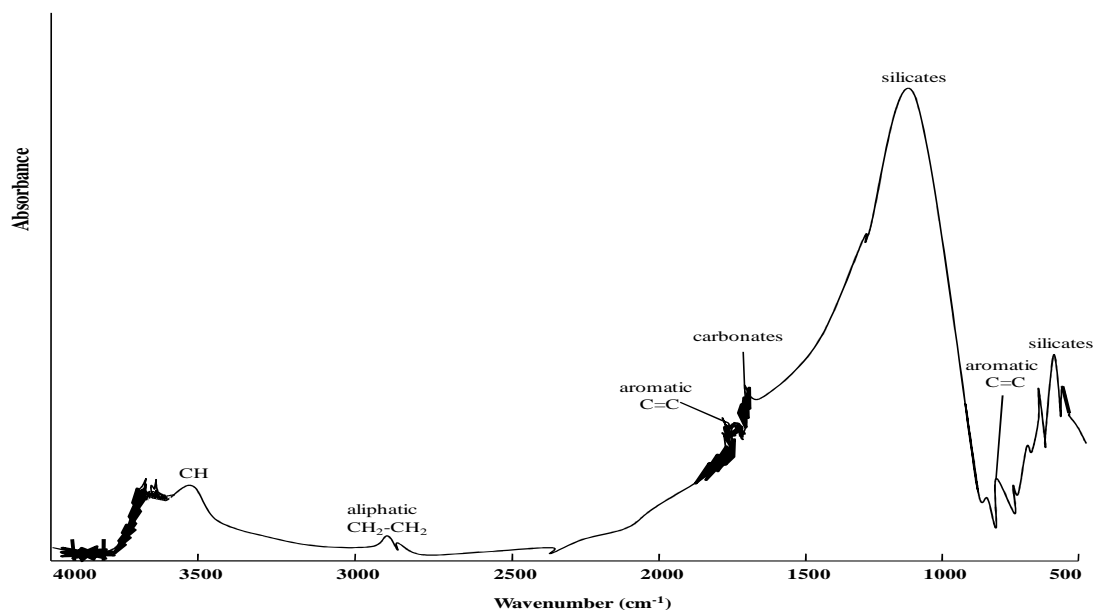


Fig. 3: FT-IR spectrum of shale from the Anambra Basin, showing the principal functional groups related to organic matter between 4000 cm^{-1} and 400 cm^{-1} , with the most prominent band that peaks at 1030 cm^{-1} attributed to silicate minerals in the bulk powder sample

Fluorescence observation showed a fairly well fluorescing liptodetrinite and alginate, with orange to yellow fluorescence. The kerogen content of 1.10 $\text{mgHC g}^{-1}\text{rock}$ is described as good, with an S_2/S_3 of 2.19 indicative of gas-prone organic matter. Random reflectance of 50 particles of vitrinite yielded a value of 0.56% R_o , consistent with its T_{max} of 430°C, all indicative of early maturity. The extractable organic matter (eom) to total organic matter (TOM) ratio of 1.87 $\text{g } 100\text{g}^{-1}$ is less than the threshold (2 $\text{g } 100\text{g}^{-1}$ organic matter) thought to be necessarily below for expulsion, while the S_1/TOC of 0.02 indicates early generation of petroleum. The hydrogen index (HI) is low compared with liptinite-rich rocks, with values slightly above 50 $\text{mg g}^{-1}\text{TOC}$ for Type III kerogen at the immature stage. The oxygen index (OI) is high, suggesting deposition in a high oxygen environment and high terrestrial higher plant contribution.

Rock-Eval prolysis yields parameters that are used to describe the generation potential of a source rock by providing information on organic matter quality, type and maturity, with the TOC, S_2 and HI as relevant parameters [Peters, 1986]. The HI of 41.20 $\text{mgHC g}^{-1}\text{TOC}$ of this shale is low and results to a Type III kerogen at early maturity stage. The gas-prone nature of this rock rules out Type II kerogen, which usually shows S_2/S_3 greater than 5, while the maturity from vitrinite reflectance as well as T_{max} suggest that the current HI results from thermal evolution of a Type III kerogen, with initial HI between 600 $\text{mgHC g}^{-1}\text{TOC}$ and 850 $\text{mgHC g}^{-1}\text{TOC}$ [Lafargue *et al.*, 1998]. The ratio of eom to TOM of 1.87 $\text{g } 100\text{g}^{-1}$ organic matter is lower than the sorption threshold of 2 $\text{g } 100\text{g}^{-1}$ organic matter below which expulsion is considered to occur in source rocks. Esemé *et al.* (2006) also studied a shale sample in the Mamfe basin with the ratio of eom to TOM of 5.27 $\text{g } 100\text{g}^{-1}$ exceeding a standard threshold of 2 $\text{g } 100\text{g}^{-1}$ considered for expulsion to occur in organic matter. *n*-alkanes are used as a proxy for source contribution to organic matter, including algae, macrophytes and land plants [Meyers, 2003]. Their distribution in algae is dominated by low molecular weight compounds, especially *n*- C_{17} , while high molecular weight *n*-alkanes dominated by *n*- C_{29} or *n*- C_{31} are assigned to terrestrial higher plants [Tissot and Welte, 1984; Hunt, 1996]. The *n*-alkane distribution pattern support contribution from Type III kerogen because of the presence of high molecular weight *n*-alkanes considered as markers for terrestrial plant contribution [Uzoegbu, 2010]. The *n*-alkanes that constitute the prominent peaks are dominated by *n*- C_{29} , with a pattern similar to the Scotian shale.

Fig. 4 shows the chromatogram of the saturate fraction of the shale. The *n*-alkane distribution is dominated by high molecular weight *n*-alkane, with maximum at *n*- C_{29} . Ratio calculated from the chromatogram includes a pristane (pr)/phytane (ph) ratio of 6.50. The gammacerane index suggests deposition in a high oxic environment. The common redox proxy (pr/ph) is affected by source organism but its value of 6.50 also indicates major contribution from terrestrial higher plants. Pr/ph is commonly used as a redox proxy but it is influenced by source organisms and maturity [Peters and Moldowan, 1993]. Its value of 6.50 in Table 1 is present in other marine source rocks, such as the Scotian shale [Powell, 1982]. Gammacerane represents a marker for high salinity during deposition [Peters and Moldowan, 1993]. The index of 9.0 in this rock suggests deposition in very low saline waters. Salinity increases in the water column favours stratification and preservation of organic matter due to a reduced oxygen, which is

unfavourable to benthic organisms in the bottom water [Schreiber *et al.*, 1986]. Esemé *et al.* (2002) used geochemistry of brines from the Mamfe basin to suggest a genetic relation between evaporates and shales due to the correlation of sulphides found in the Mamfe basin shale by Le Fur (1965) with major ions that form the dominantly halite-evaporite. A signal for oxic conditions during deposition, such as low homohopane index, was found. However, the consistency of different independent parameters, such as the high oxygen index and pr/ph ratio – though affected by source and maturity, together with low gammacerane – suggests deposition in an oxidizing environment.

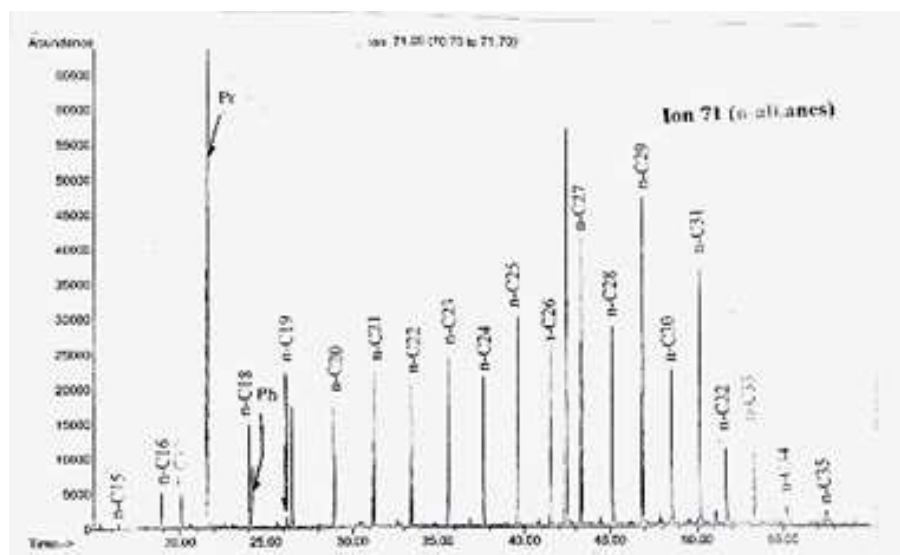


Fig. 4: Gas chromatogram of the saturate fraction, with the dominant n-alkane, pristane (pr) and phytane (ph) labeled

Maturity indicators

The production index (PI) is used to assess the generation status of source rocks but is often useful when homogeneous source rocks of different rank are compared, in which case it is characterized as the transformation ratio [Bordenave *et al.*, 1993]. Hunt (1996) suggested that a PI from 0.08 to 0.4 is characteristic of source rocks in the oil window. The value of 0.06 of this shale is consistent with its vitrinite reflectance of 0.56% R_o . This maturity is also consistent with the fairly well fluorescing organic matter as well as Rock Eval T_{max} of 430°C, reaching the 430–435°C for low sulphur immature source rocks containing Type III [Bordenave *et al.*, 1993; Hunt, 1996]. The PI is not affected by expulsion [Rullkötter *et al.*, 1988] and this will not limit its use as an indicator of the organic matter transformation because generation may start for rocks with Type II at 0.55% R_o (Leythaeuser *et al.*, 1980). Rullkötter *et al.* (1988) used a mass balance scheme to show that, at 0.68% R_o , the transformation ratio in the Posidonia shale from northern Germany had reached 30%.

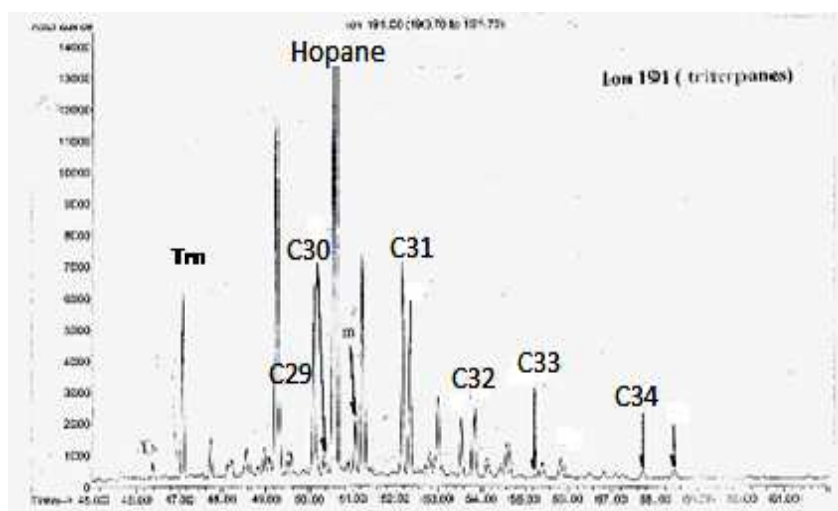


Fig. 5: Mass fragmentogram of the non-adduct fraction, showing distribution of hopane at m/z 191

The CPI of 1.67 supports the early maturity indices discussed above, though this parameter is usually high in marine source rocks that produce mostly high molecular weight hydrocarbons [Peters and Moldowan, 1993]. Fig. 5 shows the distribution of hopanes investigated at m/z 191 of the non-adduct fraction. The $17\alpha\beta$ - C_{30} hopane is dominant, but also prominent is gammacerane and a step-like decrease in homohopanes up to C_{34} . The ratio 0.77 of the geo-epimer to the sum of the geo and bio-epimers of homohopane (C_{31} S/S+R) is indicative of equilibrium and support the early maturity of this shale. The ratio of the geo-epimer (C_{31} S) to the sum of the geo and bio-epimer (C_{31} R) reaches equilibrium around 0.76 [Radke *et al.*, 1977], corresponding to the main stage of diagenesis [Peters and Moldowan, 1993]. More mature samples will be revealed by drilling and the volume of source rocks will be crucial in the evaluation of their potential to generate commercial accumulations. Various maturity indicators suggest that this shale is at the onset of oil window and its current HI of $41.20 \text{ mgHCg}^{-1} \text{ TOC}$ is thought to reflect thermal evolution due to labile kerogen from an initial HI between $600 \text{ mgHC g}^{-1} \text{ TOC}$ and $850 \text{ mgHCg}^{-1} \text{ TOC}$, characteristics of Type III kerogens [Lafargue *et al.*, 1998].

POTENTIAL PETROLEUM SYSTEMS

The buildup of any prospect or of a petroleum system requires the availability of good-quality source rocks. Additionally, the stratigraphic position of the source rocks, the availability of good-quality reservoir and seal lithologies, timing of hydrocarbon generation, favourable regional migration pathways, and trapping mechanisms must also be considered. Anambra basin has the best natural petroleum system in terms of formational source rock, reservoir, and seal lithologies. Apart from interbedded marine sandstone facies in the Nkporo/Enugu shales, petroleum generated from the Nkporo/Enugu shale and Mamu coal source rocks have good reservoirs in the overlying clean quartz arenites of the Ajali sandstone. Sandstones of the Mamu Formation could provide additional reservoirs. The overlying shales of the Imo and Nsukka formations are expected to provide regional seal for hydrocarbon that may have been generated and migrated into reservoirs in this part of the Benue trough. Because the intense Santonian deformation had occurred prior to the deposition of sediments and hydrocarbon generation in this basin, only the well documented abrupt facies changes would likely provide stratigraphic traps for hydrocarbon accumulations in this basin. However, local Cenozoic tectonics could also have enhanced the formation of some structural traps [Obaje *et al.*, 2004].

CONCLUSION

Shale from the Anambra basin has been characterized for its source potential using bulk and molecular geochemistry. The shale is a good quality source rock, with gas-prone kerogen. The high oxygen index, high pristane/phytane ratio, low gammacerane index, as well as distribution of organic matter-related functional groups on its infrared spectrum, suggest deposition in an oxidizing environment. In addition to the functional group distribution on the infrared spectrum, the n-alkane distribution, dominated by high molecular weight compounds, together with the hydrogen index, is considered to be characteristic of Type III kerogen. Multiple indicators, including R_o , CPI, T_{max} and C_{31} homohopane ratio, consistently indicate an early mature source rock. Generated petroleum may not yet have reached the threshold for hydrocarbon expulsion and a review of petroleum system elements in the basin may stimulate more interest in exploration in the area.

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