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ABSTRACT

The drive for successful hydrocarbon exploration and production in the inland sedimentary basins of Nigeria necessitates detailed evaluation of the paleoenvironments and generative potential of the source rock facies of the Campanian to Maastrichtian Enugu Formation exposed around Enugu, Anambra Basin.

The Anambra Basin, located in the southern Benue Trough is a post Santonian synclinal sedimentary structure containing over 5,000m thick of upper Cretaceous to recent sediments. The investigated sections consist of coarsening upward sequences with thick, dark gray shale at the base, grading upward through siltstones into thin, texturally mature sandstone. The lithologic assemblage suggests deltaic progadation during active delta growth. The carbonaceous shale at the base is interpreted as full marine pro-delta subfacies while the upper part of the section represents shallow water shoreface subfacies.

The total organic carbon (TOC) values for the source rock intervals vary from 1.29 to 4.42wt. % (average = 2.85wt.%) with all the samples exceeding 0.5wt%. Soluble organic matter content (SOM) ranges from 578 to 1931ppm (average = 997). These suggest fair to good source rocks in the sections studied. The low hydrogen index (HI) values (29 to 128mgHC/g TOC, average = 70) and predominance of vitrinite reveal prevalence of terrestrially derived type III organic matter in the samples and their potential to generate gas.

Most of the samples studied exhibit above-average hydrocarbon pyrolytic yields ($S_1, S_2\geq2,000$ppm hydrocarbon) and therefore can be considered moderate to fair hydrocarbon source. However, the Tmax values which range from 425 to 434°C and Bitumen to TOC ratio ranging from 16.38 to 72.11mg ext./g TOC and the spore/pollen colour index suggest low thermal maturation levels for active source rocks.

(Keywords: hydrocarbon exploration, total organic carbon, TOC, soluble organic matter, SOM)

INTRODUCTION

The Anambra Basin is located in the southern part of the regionally extensive northeast-southwest trending Benue Trough (Figure 1). It is a synclinal structure consisting of more than 5,000m thick of Upper Cretaceous to Recent sediments representing the third phase of marine sedimentation in the Benue Trough (Ladipo, 1988; Akande and Erdtmann, 1998).

Reports of various authors indicated that the basins evolved consequent to the Late Jurassic to Cretaceous basement fragmentation, block faulting, subsidence, rifting and drifting apart of the South American and African plates and therefore representing part of the West African Rift Systems (WARS) (Fairhead and Okereke, 1987; Genik, 1992). Maurin et al. (1986) and Benkhelil (1989) interpreted the Benue Trough system as a set of pull-apart basins generated by sinistral wrenching along pre-existing NE-SW transcurrent faults.

The stratigraphic succession of the Anambra Basin comprises of the Campanian to Maastrichtian Enugu/Nkporo/Owelli formations (lateral equivalents). This is succeeded by the Maastrichtian Mamu and Ajali formations. The sequence is capped by the Tertiary Nsukka Formation and Imo Shale (Figure 1). The detailed stratigraphic description is available in several publications (Petters, 1978; Agagu et al. 1985; Reijers, 1996).
The paleoenvironments, biostratigraphy and petroleum geology of the Anambra Basin have attracted the attention of many authors. Agagu and Ekweozor (1982) reported that the Awgu and Nkporo shales constitute the main source and seal rocks in the Anambra Basin. Ekweozor and Gormly (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. The work of Unomah and Ekweozor (1993) revealed that the organofacies of the Nkporo Shale are provincial with the Calabar Flank having the highest oil potential whereas those in the

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Anambra Basin and Afikpo Syncline are gas prone. According to Akande et al. (1992), the lower Maastrichtian Coals of the Mamu Formation are characterized by moderate to high concentrations of huminite and some minor amounts of inertinites and liptinites. Akaegbobi and Schmitt (1998) supported the earlier reports; that the Nkporo Shale is dominated by type III/II Kerogens with dominance of terrestrially derived organic matter in the Anambra Basin.

Other studies on paleoenvironments and biostratigraphy include Gebhardt (1998) who suggested that benthic foraminiferal assemblage from the lower Mamu Formation at Leru represents deposits of pro-delta to lagoon environments. Mode (1991) dated the Nkporo Shale, south of Leru, Maastrichtian. There are other outcrop based studies of the Anambra Basin which suggest predominant influence of marginal marine in the Nkporo, Enugu and Mamu formations (Ladipo, 1988; Nwajide and Reijers, 1996).

From the above, it is obvious that most of the previous investigations focused mainly on the Nkporo and Mamu Formations. Even the recent work of Obaje et al. (2004) on the hydrocarbon prospectivity of Nigeria's inland basins did not provide organic geochemical information on the Enugu Formation which is well and extensively exposed in Enugu.

The objectives of the present study therefore include; (i) description of surface lithologic sections of the Enugu Formation, their locations, biogenic and physical characteristics and reconstruction of their depositional processes (ii) determination of the quantity and quality of the source rock facies (iii) evaluation of the thermal maturity and hydrocarbon generation potential of the organic rich sediments by means of organic petrological and geochemical methods.

**ANALYTICAL METHODS**

This study involved measurements, documentation of sedimentologic features and sampling of two surface lithologic sections of the Enugu Formation at Enugu. Fresh samples of shale and siltstone were selected for series of organic geochemical, petrological and palynofacies analyses.

**Total Organic Carbon (TOC) Determination**

Thirty five shale samples were selected for preliminary total organic carbon content determination using Walkley Black wet oxidation method. 0.5g of each of the pulverized sample were subjected to chromic oxidation following the procedure of Walkley and Black (1965). This assessment served as preliminary screening for further detailed Rock-Eval analysis and to determine the organic richness of the source rocks.

**Rock-Eval Pyrolysis**

Twenty nine screened samples were further analysed by Rock-Eval pyrolysis to determine the hydrocarbon generation potential, maturity, type of kerogen and hydrogen richness (HI) using a Rock-Eval II pyrolyser machine with TOC module at Humble Geochemical Services Division, Texas, USA. The samples were heated in an inert atmosphere to 550°C using a special temperature programme.

The samples were heated to a temperature of 300°C for 3 min to generate the first peak (S1) which represents free and adsorptive hydrocarbon present in the sample. This was followed by programmed pyrolysis to 550°C at 25°C/Min. The second peak (S2) represents the hydrocarbon generated by the thermal cracking of the kerogen. At the same time, the CO2 produced during the temperature interval was recorded as the S3 peak. Other parameters obtained from the instrument include Tmax, that is temperature corresponding to the maximum S2 peak, Hydrogen Index (HI) and Production Index (PI).

**Determination of Soluble Organic Matter (SOM)**

The main reagent used for the soluble organic matter extraction was dichloromethane. The samples were extracted in standard soxhlet extractor. Refluxing was done for 24 hours to ensure exhaustive extraction. The bitumen were then filtered at room temperature and weighed in a clean glass bottle. The weighed extract was measured in parts per million (ppm). The analysis was carried out at the Institute of Agricultural Research and Training (IAR&T), Ibadan.
Organic Petrology and Palynofacies Analysis

In order to determine the maceral constituents of the shales, eight selected samples were crushed to less than 2mm and impregnated in epoxy resin, ground and polished for quantitative reflected light microscopy at the Department of Geology, University of Ilorin. Microscopic examination was carried out under X40 oil immersion objective.

For the spore/pollen analysis, ten samples were selected for palynological preparation. The samples were prepared according to standard palynological procedures involving acid treatments (HCl and HF) and sieving. The slides were carefully studied using Leitz Diaplan microscope for identification and documentation at the Institut fur Angewandte Geowissenschaften of the Technical University, Berlin.

Lithofacies Description and Depositional Environments

Two outcrop sections of the Campanian to Maastrichtian Enugu Formation near Milikhen hill, along Onisha-Enugu highway and at Km1, Enugu-Port Harcourt highway (Figure 1) were studied. The Enugu Formation section near the Milikhen Hill is about 50m thick and consists of shale, siltstone, and sandstone (Figure 2). The shale which forms the prominent lithology in the section is dark gray to black in colour, fossiliferous, fissile and micaceous. The shale, with average thickness of 3m, is rhythmically interbedded with relatively thin ironstone beds or ferruginised siltstones (Figure 3).

The ironstones are commonly concretional and in places wavy to parallel laminated. In most part of the section, the shale is fractured vertically and the fractures are frequently filled with ferruginised silty and clayey materials. At an interval, towards the top of the section, the shale is displaced and juxtaposed with a heteroliths of sands and silts into fine grained sandstone (Figure 3). The shale commonly grades upward into siltstone and sandstone. The siltstone show laminations (parallel to wavy), however, the lamination is distorted in places by Thallasinoides and Ophiomorpha burrows. A lot of well preserved large vegetal remains are contained in the siltstones. The fine grained sandstone is friable to slightly lithified, clean, compositionally mature and well sorted. Wave ripples, cross and parallel laminations and bioturbations are common sedimentary structures preserved in the sandstone.

A vertical facies analysis of the entire section indicates a coarsening upward vertical relationship. Two coarsening upward cycles are recognized in this section with each cycle beginning with grey to dark gray shale at the base passing upward through siltstone or heteroliths of sands and silts into fine grained sandstone (Figures 2 and 3). The Enugu Formation section studied at Garaki, Km1, along Enugu-Port-Harcourt highway is about 30m thick. It consists of similar lithologies as in the one described above. The sequence also coarsens upward with shale at the base passing through siltstone into sandstone (Figure 4).

The lithologic assemblage suggests possible deltaic progradation during active delta growth. The fossiliferous, carbonaceous shales at the base represent full marine prodelta subfacies. This shallows upward into tidally influenced shoreface and marshes. Reijers (1986), based on the sideritic nature of the shales, suggest an open marine setting for the basal shales. Kolawole (2004) and Ojo et al. (2003) reported some benthic foraminifera genera Gavelinella, Planulina and Cibicides from the shales in the studied sections which are known to be shelf environment dwellers (Peters, 1983).

Organic Geochemistry and Petrology Data and Interpretation

Several authors have demonstrated the usefulness of organic geochemical and organic petrologic methods in assessing the generative potential and characteristics of source rocks (Peters 1986; Baskin, 1997; Akande et al. 1998; Ojo and Akande, 2002). In this study, the quantity, quality and thermal maturity of the studied samples of the Enugu Formation are discussed based on Rock-Eval pyrolysis data (Tmax, TOC, HI, PI and SP) and petrographic data.

Organic Richness

Petroleum is a generative product of organic matter disseminated in the shale and therefore the quantity of hydrocarbon directly correlates with organic matter concentration of the potential source rocks (Tissot and Welte, 1984).
Figure 2: Lithologic Section and Geochemical Log of the Enugu Formation exposed near Milikhen Hill, along Enugu – Onitsha Expressway, Enugu.
Figure 3(a): Thick Carbonaceous Shale at the base of the Enugu Formation exposure near Milikhen Hill. Note the thin ferruginous interbeds. (b) Middle Part of the Exposure. Note the shale which grades up into sandstone.
### Figure 4: Lithologic Section and Geochemical Log of the Enugu Formation Exposed at Gariki, Km 1, along Enugu - Port Harcourt Expressway.

<table>
<thead>
<tr>
<th>Thickness (m)</th>
<th>Lithology</th>
<th>Sample No.</th>
<th>TOC (Wt. %)</th>
<th>S1 + S2 (mg HC/g rock)</th>
<th>T&lt;sub&gt;max&lt;/sub&gt; (°C)</th>
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<td>5</td>
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<td>EP1L, EP1J</td>
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<table>
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<tr>
<th>Thickness (m)</th>
<th>Lithology</th>
<th>Sample No.</th>
<th>TOC (Wt. %)</th>
<th>S1 + S2 (mg HC/g rock)</th>
<th>T&lt;sub&gt;max&lt;/sub&gt; (°C)</th>
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</table>
The amount of organic carbon is usually measured as total organic carbon (TOC). The TOC of the shale samples ranges from 1.29 to 4.42 wt.% with an average of 2.85 wt.% in 29 samples (Table 1). The average TOC value in the sections indicates a moderate to high organic matter concentration (Herdberg and Moody, 1979; Hunt, 1979; Peters and Cassa, 1994).

Most of the samples have TOC values in excess of 2.0 wt.% and such levels of organic enrichment are considered one of the necessary prerequisites for a rock to be classified as effective hydrocarbon source rock worldwide. Although the lithology of the investigated sections is quite monotonous, a vertical variation in the TOC values, which increases up section is observed (Figures 2 and 4).

The source rock facies at the lower part of the section, representing probably the pro-delta facies have lower concentration of organic matter. The upper part of the section representing the proximal part of the delta are richer in organic matter. The probable factors responsible for this may be attributed to localized changes in biological productivity, proximity to organic sources and preservation condition (Bustin and Chonchawalit, 1997; Ojo and Akande, 2002).

### Table 1: Organic Geochemical Data (Rock-Eval Pyrolysis and Soluble Organic Matter) from Enugu Formation Samples.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Location</th>
<th>TOC (wt.%)</th>
<th>SOM (PPM)</th>
<th>SOM/TOC (mg ext./STOC)</th>
<th>S1 (mgHC/g rock)</th>
<th>S2 (mgHC/g rock)</th>
<th>SP (S1+S2)</th>
<th>Tmax (°C)</th>
<th>PI</th>
<th>H1 (mgHC/g TOC)</th>
<th>Type of Kerogen</th>
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</tbody>
</table>
The suggestion of anoxia at the basal part of the section at Milikhen Hill, proposed by Reijers (1996) based on the dark colour of the shales is not supported by the present geochemical data. The soluble organic matter (SOM) values were obtained for the samples to further assess their organic matter concentration (Idowu et al. 1993; Baker, 1972). The SOM values vary from 578 to 1931ppm (Table 1) with average of 997ppm in the two sections studied. These values indicate moderate to good concentration of organic matter and therefore fall within the range of adequate source rocks (Dow, 1977; Unomah and Ekweozor, 1993).

**Types and Quality of Organic Matter**

The relatively low hydrogen index (HI) values of the studied samples which range from 29mgHC/gTOC to 128mgHC/gTOC (Table 1) suggest that the source rocks have potential for gas. The most significant factor with respect to the capacity of source rocks to generate petroleum is the amount of hydrogen in the kerogen (Hunt, 1996). Hydrogen-rich organic matter commonly generates more oil than hydrogen poor organic matter because oil is rich in hydrogen. According to Baskin’s (1997) classification, source rock with HI <100mgHC/gTOC is gas prone, 100-200mgHC/gTOC is gas and oil prone and; >300mgHC/gTOC is classified as oil source rock. Gas prone source rocks are typical of type III kerogen.

With the substantial volume of the source rocks in the study area, coupled with the obtained average HI value of more than 20mgHC/gTOC, the shales of the Enugu Formation will be effective source of gaseous hydrocarbon in the Anambra Basin (Killops and Killops, 1993). The plot of HI versus Tmax (Figure 5) confirms the predominance of type III organic matter.

It should be noted that low HI index values reported in this study is comparable with low trend of HI reported in the Campanian to Maastrichtian source rock facies (except the Mamu Formation Coal) in the Nigerian sedimentary basins (Obaje et al. 2004; Ehinola et al. 2005; Akande et al. 2005). This is an indication of significant contribution of organic materials from terrestrial sources in the rift basins during the upper Cretaceous in Nigeria.

![Figure 5: Classification of Kerogens of the Enugu Formation on HI – Tmax Diagram.](image-url)
A comparative assessment, to further define the quality of organic matter in the source rocks, through maceral analysis indicates the prevalence of vitrinite maceral group. The vitrinite maceral group fluctuates between 40 to 62%. The inertinites range from 24 to 50% while the liptinites range from 12 to 19%. The vitrinite components are mainly desmocollinite, collinite and vitrodetrinitine. The inertinites group consists of fusinites and semifusinites whereas the liptinites components are mainly cutinites and sporinites. The inertinites and the vitrinite maceral which dominate Enugu shales are derived predominantly from structural part of plants and are deficient in hydrogen. They correspond to type III kerogen (Tissot et al. 1974; Van Krevelen; 1961, Akande et al. 1992).

**Thermal Maturity and Hydrocarbon Generation**

The organic maturation of the studied samples is evaluated based on Tmax values of the shales which range from 425 to 434°C (Table 1, Figures 2 and 4). The thermal maturity of organic matter is commonly derived from Rock-Eval Tmax, which is the pyrolysis temperature (°C) at the maximum rate of kerogen conversion (Baskin, 1997). The HI – Tmax diagram (Figure 5) indicates that all the samples analysed are in the diagenetic level of burial to early mature (Espitalie et al. 1984; Gries et al. 1994). Only seven of the twenty-nine samples with Tmax value between 430 and 434°C are in the beginning of hydrocarbon generation, others are immature. A lot of workers have indicated that Tmax values of organic matter are in good agreement with vitrinite reflectance and elemental atomic H/C and O/C ratios (Hendrix et al. 1995; Wan Hasiah, 1999; Adekeye et al. 2006).

The ratio of bitumen yields (SOM) to total organic carbon (TOC) (SOM/TOC) which has been used by many authors (Miles, 1989; Idowu and Ekweozor, 1993; Peters and Moldowan, 1993) to evaluate thermal maturity of source rocks show a pattern similar to the Tmax values in this study. The SOM to TOC ratio for the samples range from 16.3 to 72.1mg ext./g TOC, which suggests immature to marginally mature. It should be noted that the thermal maturation of some of the samples have passed the early stage of diagenesis but have not reached the beginning of enough generation of hydrocarbons and consequent initial migration (Van-denbroucke et al. 1993). There is no indication of hydrocarbon migration in the samples as the Production Index (PI) is low.

Values of the maturity indicators discussed above are consistent with the palynofacies characteristics of the studied source rocks. The colour of the miospores from the samples are characterized by light to brown and rarely dark brown which suggests low thermal condition. The relationship between the colour of spores and pollen and, petroleum generation and expulsion from kerogens have been demonstrated in several studies (Staplin, 1977; Hart, 1986). A progressive change in colour from light brown to yellow (diagenesis) through brown to dark brown (catagenesis) and finally black (metagenesis) was proposed by Tissot and Welte (1984) and is adopted in this study.

**Hydrocarbon Source Potential**

Rock-Eval Pyrolysis reveals that the total generation potential (S1+S2) of the sample fluctuates between 0.56 to 4.51mgHC/g rock (Table 1) with a mean of 2.13mg HC/g rock. S1 is the quantity of free hydrocarbon liberated by volatilization at 300°C and expressed as mgHC/g rock while S2 is the quantity of hydrocarbon produced by further thermal cracking of kerogen also expressed as mgHC/g rock. As with the total organic carbon (TOC) of the shales, most of the samples showed source potential (SP), that is, hydrocarbon pyrolytic yields exceeding minimum required for hydrocarbon source rocks (Figures 2 and 4).

The assessment is based on Tissot and Welte’s (1984) classification as follows; rocks with lower than 2mgHCg/ rock (2,000ppm) have little or no oil source rock potential but some potential for gas whereas rocks with SP from 2 to 6mgHC/g rock (2,000 to 6,000ppm) are classified as having moderate to fair oil source rock potential and some potential for gas. Those with SP greater than 6mgHC/g rock (>6,000ppm) are considered as good or excellent petroleum source rocks. In this study, majority of the samples exhibit yields consistent with moderate to fair source rock potential especially for gas (Dyman et al. 1996). The cross plot of S2 versus TOC (Figure 6) indicates that the Enugu Formation samples are secondary source rocks with potential to generate gas and condensate.
Burwood et al. (1995) defined effective primary source rocks as those having $S_2$ greater than 5mgHC/g rock and effective non source rocks (ENS) as those with $S_2$ less than 1mgHC/g rock.

CONCLUSIONS

The investigated sections of the Enugu Formation exposed at Enugu consists of coarsening upward sequences with thick carbonaceous shale at the base, passing through siltstone into thin texturally mature sandstone. They are interpreted as prodelta (open marine) to tidally influenced shoreface (shallow marine) deposits.

The source rocks are moderately to fairly rich in organic matter and can therefore be considered as potential source rocks. Rock-Eval pyrolysis and maceral analysis data reveal that Type III kerogen are prevalent, indicating substantial contribution from terrestrial source and their potential to generate gas. Most of the source rocks in the area displayed above-average hydrocarbon pyrolytic yields (average $S_1+S_2 > 2.5$ mgHC/g rock), however, thermal maturity studies indicate a low maturation level for the source rocks. At present outcrop level, hydrocarbons have not been generated.

The depositional history of the Enugu Formation favoured the development of very thick potential hydrocarbon source rocks in the proximity of potential reservoir rocks and traps. The study area is therefore considered to be of good petroleum potential particularly gaseous hydrocarbon.

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