

Oil Generation Capacity of Maastrichtian Coals from the Anambra Basin, Se NigerIA.

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---**ABSTRACT--**

Sedimentation in the Anambra Basin (SE Nigeria) thus commenced with the late Cretaceous (Campano-Maastrichtian) marine and paralic shales of the Enugu and Nkporo Formations, overlain by the coal measures of the Mamu Formation. The fluviodeltaic and subtidal sandstones of the Ajali and Owelli Formations lie on the Mamu Formation. The overlying deltaic Nsukka and marine shales of the Imo Formations were deposited during the Paleocene. These two formations (Mamu and Nsukka) consist of a cyclic succession of coals, carbonaceous shales, silty shales and siltstones interpreted as deltaic deposits. Late Cretaceous coals from the Anambra Basin region were analyzed by reflected light-fluorescence microscopy and Rock Eval 6 pyrolysis to evaluate their regional rank variation and petroleum generative potential. The coal ranks in the region range from a low of 0.56%Ro in the OKAB area to a high of 0.59%R^o in the ONYE area. Tmax (^oC) values (Rock Eval 6 pyrolysis) also increase progressively with increasing vitrinite %R^o values. However, Tmax values of coal and shale samples are between 417 and 432ºC respectively. HI values range from 21.50 to 344.53mgHC/gTOC suggesting that the samples have gas and oil-generating potential. On the basinal evaluation, incorporating source rock data from other formations in the respective sectors, plots on the modified Van Krevelen diagram and maceral data indicates excellent to fair source rock qualities (oil and gas) in the Anambra Basin.

Key words: *Maastrichtian Formations, Anambra Basin, SE Nigeria, sub-bituminous coals, source rock, oil generation.*

I. INTRODUCTION

The onshore sedimentary basins of Nigeria comprise Cretaceous and younger grabens which form part of a series of rift basins in central and west Africa. The Anambra Basin, a sub-basin in the Benue Trough, together with the neighbouring Abakaliki Basin to the east (Fig. 1), contain important coal measures which were discovered in the early part of the last century. Coal mining dates back to 1915 when sub-bituminous coals in the Mamu and Nsukka Formations (Maastrichtian, Danian: Fig. 2) were first exploited. Exploration in the coal basins also led to the discovery of extensive lignite deposits in the Oligocene–Miocene Ogwashi-Asaba Formations (Reyment, 1965). Initial mining of the sub-bituminous coals was concentrated in the Enugu area while other mines in the Owukpa and Okaba areas focussed on the Mamu Formation with the discovery of smaller deposits around Ezimo, Ogboyaga and Inyi in the middle of the last century. Total sub-bituminous coal reserves in the north-south trending coal belt were assessed to be about 1.5 billion tons (Orajaka *et al*., 1990). Lignite deposits occur in numerous seams in an east-west belt which passes through Ihioma and Azagba Ogwashi to the SW of the coal belt (Fig. 1). Lignites reserves were estimated to total about 300 million tons but are largely unexploited (Orajaka *et al*., 1990). Subbituminous coal production, mainly in the Enugu area, reached an annual output of ca. 700,000 tons in the 1980s (Orajaka *et al*., 1990), but has since declined until recently, when efforts to revive it have been proposed by the Nigerian Government. This has encouraged detailed studies of the geology and physical and chemical characteristics of the coals.

The Anambra Basin is bounded to the south by the northern portion of the Niger Delta petroleum province (Fig. 1). Eneogwe and Ekundayo (2003) identified three oil families (A, B, C) in the NW Niger Delta. Oils in family "C" were generated from source rocks with abundant terrestrial organic matter (OM), while family "B" oils were derived from source rocks with mixed terrestrial and marine OM. The petroleum system in the northern portion of the Niger Delta contains light, waxy oils sourced mainly from landplant derived OM disseminated throughout the Eocene source rock units (Bustin, 1988; Ekeweozor and Daukoru, 1994). Furthermore, onshore in the rift complex, commercial hydrocarbon accumulations have been discovered to the north of the Benue Trough in Chad and the Sudan, and gas and oil shows have also been reported in Niger (Obaje *et al*., 2004). As a part of an on-going study of the evolution of the Nigerian coal basins and the burial histories of the Cretaceous–Tertiary sedimentary succession, we have

Fig. 1: Generalised geological map of the SE Nigeria (boxed areas of inset) showing the location of the coal deposits. Numbers indicate Cretaceous and Tertiary formations 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 Akande et al., 2007).

carried out detailed petrographic analyses of 87 coal samples collected during field work between 2004 and 2006. Samples came from the Enugu- Onyeama, Okpara, Ezimo and Okaba mines.

In this paper we assess coals from four regions in the Anambra Basin (Fig. 1), describing coals in terms of petrography (macerals, petroleum content), rank (i.e. random per cent vitrinite reflectance in oil ($\%R_{o}$) and Rock-Eval 6 pyrolysis properties (e.g maximum temperature (T*max*), total organic carbon (TOC), free oil content (S1) and hydrocarbon content (S2). The samples showed good evidence for the presence of liquid hydrocarbons associated with both maceral groups and authigenic mineral matter. The purpose of this paper is to ascertain their organic matter source input, maturity and petroleum generation potentials.

II. REGIONAL STRATIGRAPHIC SETTING

Sub-bituminous coals are restricted to the Maastrichtian Mamu and Nsukka Formations which crops out in long narrow ridges in the NE–SW trending Anambra Basin (Fig.1). The Mamu and Nsukka Formations consist of alternating sandstones, sandy shales and mudstones with interbedded coal seams. The formation is underlain by the Campanian Enugu/Nkporo Shales (lateral equivalents), and overlain by the Ajali Sandstone (Middle Maastrichtian) and Nsukka Formation (Upper Maastrichtian to Danian) (Fig. 2). Five sedimentary units are recognized in the Mamu Formation in the Enugu area, where the thickest exposed section (approximately 80 m) occurs (Simpsons, 1954; Reyment, 1965). From the base, the units consist of (i) a basal shale or sandy shale, (ii) sandstones with occasional shale beds, (iii) carbonaceous shales, (iv) coals and (v) sandy shales. The Nsukka Formation has well exposed section at Iyinzu in Ezimo area with thickness of about 55 m. The succession is paralic as demonstrated by the alternating marine and

Fig. 3. Sedimentary succession at the Enugu-Onyeama Mine.

continental facies, and is considered to represent part of the "third marine cycle" in southern Nigeria (Short and Stauble, 1967; Hogue, 1977; Agagu, 1978). Sediments of the third marine cycle were mainly deposited in Campanian – Maastrichtian times in the Anambra and Afikpo Basins (Fig.1). These two post-Santonian basins were formed as successors to the Benue Trough, where deposition of the first and second marine depositional cycles took place in Albian – Santonian times (Figs 1 and 2). The third-cycle sediments were deposited during the initial growth of the proto-Niger Delta in the Late Cretaceous (Allen, 1965; Short and Stauble, 1967). An Early Paleocene transgression led to the termination of the proto-Niger Delta and the deposition of Imo Shale, which was succeeded by the regressive phases of the Ameki and Ogwashi Asaba Formations.

III. LITHOSTRATIGRAPHY

Sections through the Mamu Formation occur at the Enugu-Onyeama, Owukpa and Okaba coal mines (Figs 3, 4, 5). In general, the coal-bearing sections in these locations consist of cyclic successions of coal, carbonaceous shales, heteroliths (sandy shales and shaly sandstones), siltstones and bioturbated sandstones. The cyclothems are well exposed at the Onyeama section (Fig. 3), and are interpreted as deposits typical of a deltaic setting. At Onyeama (Fig. 3), the exposed succession shows a basal coal seam 1.2 m thick, overlain by a carbonaceous and parallellaminated grey shale. This is overlain by a heterolithic unit of grey shale with sandstone streaks, which grades upward into fine-grained sandstones. Two similar successions follow above in the 39.9 m thick section which includes three coal seams. The middle unit starts with a 1.2 m thick coal seam and contains three thick sandstone beds overlain by carbonaceous grey shales. The third 2.75 m thick coal seam occurs above. Siltstone beds cap the section.

Fig. 5. Sedimentary succession at the Ezimo Mine.

IV. MATERIALS AND METHODS

Lignites and sub-bituminous coals are widely distributed within the "coal measures" of the Maastrichitian Mamu and Nsukka formations to Miocene Ogwashi-Asaba Formations in the Lower Benue Trough (Akande et al., 1992). The coal deposits in the Lower Benue Trough occur mainly in the Enugu province where four mines: Iva valley, Onyeama, Okpara and Ribadu (Fig. 1) are being worked by the Nigerian Coal Corporation. Other coal deposits that are being worked in this area include those at Okaba, Owukpa, Ogboyaga and Ezimo; all found within the Mamu and Nsukka Formations.

A total of 87 coal samples were subjected to laboratory analyses, 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^oC 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 CO_2 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 CO_2 . 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 $4000 - 400 \text{ cm}^{-1}$. 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 characterisation.

V. RESULTS AND DISCUSSION

Kerogen Types

The average maceral composition based on the average percentages of huminite-liptinite inertinite (H:L:I) ratios are: 72:11:10 for the Onyeama coals, 69:13:12 for the Ezimo coals, 76:6:13 for the Okpara coals and 66:27:4 for the Okaba coals. This indicates a predominance of huminite (Type III kerogen) with contributions of inertinite (Type IV kerogen) and liptinite (Type II kerogen) in the organic matter.

Petrology

Random reflectance in oil of collotelinite or collodetrinite ranges from a low of 0.56%R_o for the Okaba (OKAB) coals to a maximum of $0.59\%R_0$ for the Onyeama (ONYE) coals in the southern part of the studied area (Fig.1, Table 1). Based on the vitrinite reflectance, coal ranks vary from sub-bituminous C to B and subbituminous B to high volatile A sub-bituminous (Taylor *et al*., 1998).

Table 1: Maceral and vitrinite %Ro data for coals from the Anambra Basin southeastern Nigeria.

Volatile matter ranges from 41.39wt% to 53.94wt% dry basis (Uzoegbu, 2010), which is as expected for the coal ranks indicated by reflectance. Based on volatile matter content, all the four Anambra Basin coals should have ranks of 0.55 to 0.60% R_o , or high volatile B to A.

Maceral analyses for the coals are shown in Table 1. Vitrinite (V) is the dominant maceral group, ranging from 66% in the OKAB coal to 76% in the OKPA coal. Collotelinite is the dominant vitrinite maceral with secondary collodetrinite (Fig. 6). The inertinite (I) content is variable (4.0% to 13%), with the OKAB and ONYE coals containing only 4.0 - 10%, whereas the Ezimo (EZIM) and Okpara (OKPA) coals contain higher amounts, between 12 and 13%. In the latter coals, fusinite is dominant with significant amount of inertodetrinite. Fusinite commonly contains mineral matter (MM) (silica and carbonate) or solid bitumen in cell lumen. Liptinite (L) ranges from 6.0% to 11.0% in the OKPA and ONYE coals, and 13.0% and 27.0% in the EZIM

 Fig. 6: Photomicrographs of samples viewed under white light (left side) and fluorescence light under ultraviolet excitation (right side). (a-b) ONYE-43: Sporinite macerals in a matrix of attrinite and inertodetrinite. Sporinite display orange and brownish fluorescence while inertodetrinite does not fluoresce. (c-d) OKPA-49: Cutinite macerals in a matrix of vitrodetrinite and inertodetinite. Cutinite display brown fluorescence. (e-f) OKAB-81: Resinite macerals associated with corpocollinite in a matrix of vitrodetrinite. Resinite display yellow fluorescence. (g-h) EZIM-75: Same as (e-f) though from different sample field.

and OKAB coals, respectively. Liptodetrinite, resinite, sporinite and cutinite are the most significant liptinite macerals (Fig. 6). Alginites are rare and were only noted in a few samples from the ONYE, OKPA and OKAB coals.

Based on general maceral composition, it can be concluded that all the coals were deposited in a deltaic/shallow-marine environment which is consistent with the sedimentological result obtained by Ladipo, 1988 and geochemical interpretation based on very low pyretic sulphur 0.50% - 0.98wt% and very low concentrations of organics (Uzoegbu, 2010) generally supported the aforementioned depositional environment. The maceral collodetrinite is mostly associated with liptinites in all the coal seams, and is considered to have enhanced liquid petroleum generation potential (Mukhopadyay and Hatcher, 1993). A minimum content of 15- 20% liptinite is considered to be the threshold for a coal to be considered as a potential petroleum source rock (Hunt, 1991; Fowler *et al*., 1991).

Rock-Eval 6 Pyrolysis

Rock Eval 6 pyrolysis data are shown in Table 2 and plots of the coals as kerogen type are shown in Fig. 7. All of the samples have Hydrogen Indices (HI) on the order expected for oil and gas prone humic coals, although the oxygen indices are lower than expected for normal humic coals. Based on the Mukhopadyay and Hatcher (1993) classification of kerogen in coals relative to HI and OI, all of the samples plot between Types II and III, or within the mixinitic field, where desmocollinite (collodetrinite) is associated with liptinites. good potential for the generation of oil/gas. This type of coal facies is considered to have enhanced liquid petroleum generative potential (Mukhopadyay and Hatcher, 1993); a minimum content of 15-20% liptinite is considered to be the threshold for a coal to be considered as a potential petroleum source rock

Table 2: Rock-Eval pyrolysis data for samples from Cretaceous formations from the Anambra Basin, Nigeria.

Table 2: Continued.

(Hunt, 1991; Fowler *et al*., 1991). S1 values are relatively low for coals of this rank and composition (Sykes and Snowdon, 2002). The cause of these low values is not known but may be due to expulsion or oxidation which can reduce S1 by up to 50% (e.g. Sykes and Dow, 2001).

OI (mgCO2gTOC)

Fig. 7: HI versus OI plots of Cretaceous coal samples from the Anambra Basin.

On the selected samples for Rock-Eval 6 and organic petrography S1 ranges from 0.26 in ONYE to 3.52mgHC/g rock in OKAB while S2 ranges from 124.94 in OKPA to 240.07mgHC/g rock in ONYE. HI ranges from 180.21 in EZIM to 344.50mgS2/gTOC in ONYE and (S1+S2)/gTOC ranges from 126.28 OKPA to 242.04 in ONYE. Vitrinite reflectance $(\%R_0)$ ranges from 0.48 in EZIM to 0.59 in ONYE. These parameters [S1, S2, HI and $(S1+S2)/gTOC$] were plotted relative to %R₀.

In agreement with the petrographic composition, the HI versus OI diagram classifies the coals as containing an abundance of Type III kerogen (Fig. 7). Also, atomic H/C ratios of 0.80–0.90 and O/C ratios of 0.11–0.17 show that the coals generally plot in the upper part of the Type III band on a van Krevelen diagram. However, despite the fact that bulk geochemical analyses classify the coals as dominated by Type III kerogen, the petrographic analyses have demonstrated the presence of relatively large amounts of liptinite, such as sporinite and cutinite, in the coals. Although humic coals dominated by huminite have proven to be oil-prone (e.g. Petersen, 2006; Petersen and Nytoft, 2006), the liptinite macerals are more paraffinic in structure and they are likely to be more oil-prone than the huminite. This is favourable for the oil-proneness of the coals, as the oil expulsion efficiency is dependent on the coals' ability to generate long-chain aliphatics (>C20-25) (Isaksen *et al*., 1998).

Figs. 8-9 shows the peaks to plateaus exhibited in these plots for S1, S2, HI and $(S1 + S2)/gTOC$ at 0.58% R_o for HI, and between 0.53, 0.56 and 0.60%R_o for S1, S2 and (S1 + S2)/gTOC indicating the level of maturity and areas of hydrocarbon generation potential from $%R_0$. The peaks further shows that areas below %R_o of 0.55 will have low yield of hydrocarbon consequently have low expulsion rate. Larger data sets have been used by Sykes and Snowdon (2002) and Petersen (2002).

Fig. 8: (a) S1 (mgHC/g rock) versus vitrinite $%R_0$; (b) S2 (mgHC/g rock) versus vitrinite $%R_0$.

Similar peaks in Rock-Eval 6 parameters relative to T_{max} or vitrinite % R_0 have been noted previously in coals by Pittion and Gouadain (1985; S1/TOC peak ~1.0%Ro, T_{max} ~450°C), Suggate and Boudou (1993; HI peak at T_{max} ~440°C and $\sim 0.80\%$ R_o), Boreham *et al.* (1999; HI peak at $T_{max} \sim 440^{\circ}$ C and $\sim 0.80\%$ R_o), Petersen 2002; S2 and S1 at 0.85% R_o, HI at 0.90%Ro), and Stasiuk *et al*. (2006; HI peak ~ 0.90%Ro; between 0.90 and 1.0%R^o for peaks S2 and S1+S2/TOC). The peaks in these coals indicates that hydrocarbon generation potential increases with increasing rank, but further work is required to assess the hydrocarbon potential of the coals from these areas.

VI. 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).

VII. CONCLUSION

Coal ranks in the Anambra Basin region of southeastern Nigeria range from a low of 0.56%Ro at OKAB to a high of $0.59\%R_0$ in the ONYE area. As well as, T_{max} (°C) values (Rock-Eval 6 pyrolysis) increase with increasing vitrinite %R_o values. The classification of kerogen in these coal and shale samples relative to HI and OI, of all the samples falls between type II and type III or within the mixinitic field with good potential for the generation of oil/gas. Some indicators, such as %R_o and T_{max} consistently indicate an immature to onset of maturity and early to beginning oil expulsion. Liquid petroleum was likely generated within some of the coals as suggested by moderately high hydrogen indices, persistent and oily exudations from the coals during UV exposure, and traces of hydrocarbon fluid inclusions.

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