Aspects of the Hydrocarbon Potential of the Coals and Associated Shales and Mudstones of the Mamu Formation in Anambra Basin, Nigeria

M. A. Adeleye1*, C. Y. Ugboaja1 and L. Yuhong2
1Geology Department, University of Ibadan, Ibadan, Nigeria.
2State key laboratory of Organic Geochemistry, Guangzhou Institute of Geochemistry, China.

Abstract
Coals and associated shales and mudstones of the Mamu Formation in Anambra basin were examined for their hydrocarbon potentials by subjecting them to total organic carbon (TOC) and Rock-eval Pyrolysis analyses. The TOC results range from immature with respect to hydrocarbon generation and generally at low level conversion. The coals and the associated shales and mudstones are dominated by kerogen type III. Thermal maturity from Rock-eval data indicated that the sediments are immature with respect to hydrocarbon generation and generally at low level conversion. The coals and the associated shales and mudstones of Mamu Formation are therefore capable of generating oil and gas at appropriate maturity.

Keywords: Mamu Formation, Coals, Shales, Mudstones, Hydrocarbon generation, Kerogen, Thermal maturity

1.0 Introduction
The Cretaceous Anambra basin covers an area of about 40,000km² in the southeastern Nigeria. It is located at the southwestern part of the Benue Trough (Fig. 1). It is the structural link between the Cretaceous Benue Trough and the Tertiary Niger Delta (Agagu et al., 1985). Anambra basin is bounded by NE-SW trending fracture or flexure zones which are correlated with the onshore extensions of the Chain and Charcot fracture zones (Allix, 1987). About 3 km of Campano-Maastrichtian sediments were deposited in Anambra Basin (Agagu and Adhigije, 1983; Popoff et al., 1988). Anambra basin became a centre of attraction with the discovery of coal deposits as far back as 1905. Following the discovery of coal deposits, efforts were made to study the properties of the coal deposits, reserve estimation and appropriate exploitation method. Oil exploration also commenced in Anambra basin and underlying Abakaliki folded basin by Shell BP in 1950s leading to drilling of several exploration wells. Some studies emanating from the samples and data generated from the exploration wells have highlighted Nkporo and Mamu Formations as the main petroleum source rocks in Anambra Basin (Agagu, 1978; Ayoola and Avbovbo, 1981; Agagu and Ekweozor, 1982, Whiteman, 1982; Ekweozor and Gormly, 1983; Unomah and Ekweozor, 1993; Adeleye et al., 2016).

Prior to commencement of the hydrocarbon exploration activities in Anambra basin, the basin has been a major geological area for coal exploitation. Sub-bituminous coals occur principally at two levels within Anambra basin; the Lower coal measures (Mamu Formation) and the Upper coal measures (Nsukka Formation). Lignite deposits have also been reported from the Tertiary sediments of Ogwashi-Asaba Formation (Reyment, 1965). The Maastrichtian Mamu Formation consists of rhythmic clastic sequences of sandstones, shales, siltstones, mudstones, sandy shales with interbedded coal seams (Petters, 1978). The coal occurrences in Enugu, Okaba, Owukpa and others belong to the Lower coal measures (Mamu Formation). Earnings from exploitation of the coal deposits for energy generation and other purposes were once a major part of Nigerian income. The total reserves of the sub-bituminous coals known in major localities within Anambra basin were put at about 1.5 billion tonnes (Orajaka et al., 1990). Optimal usage of the coal for industrial applications has been guided by studies on their physical, chemical and technological properties (Orajaka et al., 1990; Ugwu, 1988). Akande et al (1992) also provided a number of industrial applications of coals. Study conducted on the composition, rank, depositional environments and technological properties of coals from southern Nigeria by Akande et al. (1992) also provided industrial applications of some coal deposits in Nigeria.

Technological advancement with respect to hydrocarbon exploration and oil-source rock correlation has shown that coal beds have been accepted as a potentially significant source of liquid hydrocarbons (Clayton, 1993; Hunt, 1991; Murchison 1987) and are increasingly becoming exploration targets in many parts of the world. For instance, coal-derived petroleum have been reported from the Adjuna and Kutei Basins, Indonesia (Kirkland et al., 1985), Junggar, Tarim and Turpan Basins, Northwestern China (Hendrix et al., 1995); Cooper Basin, Gippsland Basin, Australia; Taranaki Basin, New Zealand; (Curry et al., 1994; Kirkland et al., 1987; Johnston et al., 1991; Collier and Johnston, 1991) and Western Rift Basin, Tanzania (Mpanju, et al., 1991). In Nigeria also, efforts are increasingly directed at evaluating hydrocarbon generation potentials of various coal deposits as it applies to other countries around the world. Obaje and Hamza (2000) worked on the source rock potentials of the Awgu Formation coals in Middle Benue Trough using coal petrography method. Ehinola et al. (2002), Adedosu et al. (2012) and Obaje et al. (2004) also used biomarker and other organic geochemical methods to evaluate the source rock potentials of the Obi-Lafia and Awgu coals from the Middle Benue Trough. Coals from Mamu and Awgu
Formations in the lower Benue Trough were equally evaluated for their hydrocarbon generation potentials and depositional settings (Obaje et al., 2004; Ogala, 2011; Adedosu et al., 2011; 2014).

This study attempts to employ standard geochemical methods to evaluate parts of the hydrocarbon generative potential and thermal maturity of the coals and associated shales and mudstones of the Maastrichtian Mamu Formation exposed at Enugu, Okaba, Owukpa and Ezimo localities in the Anambra basin. This is to provide additional information on their potentials for liquid and gaseous hydrocarbon generation.

Figure 1: Map of sedimentary basins of Nigeria showing location of Anambra basin (After, Obaje 2004)

### 2.0 Geological Setting

The evolution of the Southern sedimentary basins started during Early Cretaceous with the formation of the Benue-Abakaliki Trough as a failed arm of the rift triple junction associated with the separation of the African and south American continents and subsequent opening of the South Atlantic (Burke et al., 1971; 1972; Burke and Whiteman, 1973; Fairhead and Green, 1989). The Benue Trough is a linear NE-SW trending Anticlinorium stretching from the Chad basin in the North to the Gulf of Guinea through Niger Delta in the South (Avbovbo, 1980). It is bifurcated into the upper Benue Trough, the middle Benue Trough and the lower Benue Trough (Whiteman, 1982). Although the exact areal definition of the Benue Trough as a whole has been an issue of controversy, however it is clear that it originated from a ‘pull-apart’ basin associated with the opening of the Atlantic Ocean which ended in the Early Tertiary with the development of the Tertiary Niger Delta (Petters and Ekweozor, 1982; Ekweozor and Unomah, 1990). The Santonian deformation fragmented the Lower Benue Trough into the Abakaliki Anticlinorium and the flanking Anambra and Afikpo Synclines. The Anambra basin and Afikpo Syncline began to form after the Santonian tectonic episode when the platform areas bordering the Benue Trough to the west (Anambra Platform) and to the east (Afikpo Platform) became downwarped to become depocentres respectively (Murat, 1972). Anambra basin is subdivided into a main southern Onitsha basin and a circular northern Ankpa basin which are separated by NE-SW trending Nsukka high (Agagu and Adhigije, 1983).

The stratigraphy, sedimentation and structures of Anambra basin have been described by Simpson, 1954; Reyment, 1965; Burke et al., 1972; Murat, 1972; Agagu, 1978; Petters, 1978; Agagu and Ekweozor, 1982; Petters and Ekweozor, 1982; Whiteman, 1982; Agagu and Adhigije, 1983; Agagu et al, 1985; Allix, 1987; Benkhelil, 1989; Nwajide, 1990; 2013 and many others. The stratigraphy of the Campano-Maastrichtian Anambra basin consists of the Nkporo Group (Nkporo/Enugu/Owelli Formation), Mamu Formation, Ajali Sandstones, Nsukka Formation, Imo Formation and Ameki/Bende Formation (Table 1).
The Mamu Formation of Reyment (1965), originally referred to as the Lower Coal Measures (Tattam, 1944; Simpson, 1954; Reyment and Barber, 1956) is made up of laterally variable succession of fine to medium grained well bedded sandstones, siltstones, shales, mudstones, coal seams and frequently bioturbated sandy shales. The sandstones are fine to medium grained and yellow in colour. The shales and mudstones are dark blue or grey and frequently alternate with sandstones to form a characteristically stripped rock (Whiteman, 1982). The coals are black to brownish black in colour, friable and weather rapidly (De Swardt and Cassey, 1961). The coal beds and carbonaceous shales are more concentrated in the basal section of the formation and rare towards the top. Mamu Formation displays repeated rhythmic patterns of deposition consisting of shale and sandy shale, coal occasionally with shale at the top, carbonaceous shale, sandstone with occasional shale and sandy shale or shale (De Swardt and Cassey, 1961; Whiteman, 1982). Mamu Formation sediments are shallow water deposits which were laid down as part of paralic facies of a large delta complex that also involved Ajali and Nsukka Formations (Cratchley and Jones, 1965). Coals belonging to Mamu Formation are sub bituminous in nature and they occur in the central and northern parts of the basin (Whiteman, 1982; Agagu et al., 1985). The coal seams are generally laterally impersistent and vary in thickness from few cm to about 4 m (Agagu et al., 1985). Mamu Formation has been described as paralic sequence in view of the alternating marine and continental sediments and the paleogeography of Anambra basin (Reyment, 1965; Burke et al., 1972, Murat, 1972; Petters, 1978). Agagu et al. (1985) inferred the depositional environment of Mamu Formation to be strandplain or deltaic system. The age of the formation is Maastrichtian (Simpson, 1954; Reyment, 1965). The presence of interbedded coals in Mamu Formation suggested paludal to possibly marginal marine environment of deposition (Courel et al., 1991, Ogala et al., 2009).

Table 1: Stratigraphy sequences of Anambra Basin

<table>
<thead>
<tr>
<th>PERIOD/AGE</th>
<th>FORMATION</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tertiary</strong></td>
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</tr>
<tr>
<td>Eocene</td>
<td>Bende/Ameki Formation</td>
</tr>
<tr>
<td>Paleocene</td>
<td>Imo Shale Group</td>
</tr>
<tr>
<td>Maastrichtian- Paleocene</td>
<td>Nsukka Formation</td>
</tr>
<tr>
<td><strong>Cretaceous</strong></td>
<td></td>
</tr>
<tr>
<td>Maastrichtian</td>
<td>Ajali Formation</td>
</tr>
<tr>
<td>Campano-Maastrichtian</td>
<td>Enugu/Nkporo/Owelli Formation</td>
</tr>
<tr>
<td>Santonian</td>
<td></td>
</tr>
</tbody>
</table>

3.0 Methods and Samples

Sediments belonging to Mamu Formation comprising of sandy shale, shales, coals, mudstones, siltstones and sandstones exposed at Okaba, Owukpa, Ezimo and Enugu localities of Anambra basin (Figure 2) were described and subsequently sampled for laboratory studies. At each exposure, the weathered surfaces of the lithologies were removed before lithological description and sample collection. The lithological description involves examining the rock type, colour, grain size, sedimentary structures and other attributes. Measurements of the beds thicknesses and other features were also made. The exposed sections were ultimately logged by describing the attributes of different lithologies from base to top and measuring their thicknesses and other features. Representative samples of the different lithologies were collected, properly labeled and transported to laboratory for geochemical analyses. The lithological logs of the exposed sections are shown in Figure 3. The logs revealed that the samples are essentially composed of sandy shales, shales, coals, mudstones, sandstones and siltstones. The sandy shale is fine to medium grained, fissile and grey in colour. The shales are fissile, fine grained and dark grey in colour. The coals are black in colour, fine grained, impermeable and sometimes show lamination. The coals may be fractured and the thickness ranges from about 0.2 to 1.5 m. The coals generally stain hand when touched. The mudstones are fine grained, grey to black in colour, with presence of fine, whitish sandstone or siltstone giving it a striped texture. The mudstones may also be concretionary with characteristic reddish brown colouration. The sandstones are white or yellowish brown in colour and fine to medium grained. They may be crossbedded or laminated. The siltstones are fine grained and white to light brown in colour.

The coals, shales and mudstones samples were finely pulverized stored in vials and labeled. The samples were treated with concentrated hydrochloric acid to remove carbonates and the total organic carbon (TOC) was measured on carbonate free samples using Elementar Vario EL III elemental analyser (Hanau, Germany). Using minimum value of 0.5 wt. % TOC for the samples, eighteen (18) samples were further subjected to Rock-eval pyrolysis (Rock-eval Pyrolyser II). Rock-eval data include Hydrogen Index (HI), Oxygen Index (OI), Genetic Potential (GP), Production Index (PI), maximum temperature
(Tmax) etc. Calculated vitrinite reflectance (Calc. % Ro) was generated from Tmax using the formula: Calc. % Ro = 0.0180 × Tmax (Jarvie, 1991). The Total organic carbon and Rock-eval pyrolysis analyses were carried out the State Key Laboratory of Organic Geochemistry, Guangzhou Institute of Geochemistry, China.

Figure 2: Map of Nigeria showing sample collection localities in Anambra Basin.

Figure 3: Lithological logs of the studied sections of Mamu Formation.
4.0 Results and Discussions

4.1 Organic Matter Richness

The results of the Total organic carbon (TOC) and Rock-eval Pyrolysis of the samples are shown in Table 2. The TOC values of the Mamu Formation sediments range from 0.07 to 67.68 wt. %. Tissot and Welte (1984) proposed a minimum TOC of 0.3 and 0.5 wt. % respectively for effective carbonate and shale source rocks. Peters (1986) also reported that the commonly accepted minimum TOC content for a potential source rock is 0.5%, while rocks containing less than 0.5% TOC are considered to have negligible hydrocarbon source potential. However, Jones (1987) concluded that there is no significant difference between the required minimal TOC for carbonate and non-carbonate source rocks. Peters (1986) further stated TOC values ranging between 0.5 and 1.0 % indicate marginal and more than 1.0 % TOC often has substantial source potential. TOC values between 1.0 and 2.0 % are associated with depositional environments intermediate between oxidizing and reducing, where preservation of lipid-rich organic matter with source potential for oil can occur. TOC values above 2.0 % often indicate highly reducing environment with excellent source potential. Measurement of TOC in sediments is not sufficient enough to identify potential hydrocarbon source beds because transported terrestrial organic matter from a previous sedimentary cycle can create an organic richness of about 4 wt. %, yet this concentrated organic matter could be hydrogen poor and without significant petroleum generating potential (Demaison and More, 1980; Demaison and Shibaoka, 1975; Dow, 1977).

Table 2: Results of the TOC and Rock-eval Pyrolysis of Mamu Formation sediments

<table>
<thead>
<tr>
<th>Sample LD</th>
<th>Lithology</th>
<th>TOC Wt.%</th>
<th>S1 (mg/g)</th>
<th>S2 (mg/g)</th>
<th>S3 (mg/g)</th>
<th>GP (mg/g)</th>
<th>Tmax (ºC)</th>
<th>Calc %Ro</th>
<th>HI mg/g</th>
<th>OI mg/g</th>
<th>PI</th>
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<td>432</td>
<td>0.62</td>
<td>144</td>
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The TOC results showed that the organic carbon varies from 45.56 to 67.68 wt. % in coals, 0.07 to 5.44 wt. % in the shales, and 0.12 to 4.46 wt. % in the mudstones, indicating that the sediments contain good to high organic matter contents except for two samples with TOC value below the threshold value of 0.5 wt. %. The distribution of the TOC across the lithologies shows a fair to excellent organic matter contents in the sediments. To further highlight the source rock potential of the sediments, the generative potential (GP =S1+S2) is used. Rocks having GP value < 2mg HC/g rock corresponds to gas prone rocks or non-generative rock, rocks with GP between 2 and 6 mg HC/g rock are moderate source rocks and those with GP > 6mg HC/g rock are good source rocks (Peters and Cassa, 1994; Tissot and Welte, 1984). Rock–eval Pyrolysis results showed that the shales have GP values ranging from 2.85 to 15.66 mg HC/g TOC, those of mudstones range from 0.54 to 10.96 mg HC/g TOC, while the GP values for the coals range from 114.99 to 159.54 mg HC/g TOC. These suggested that the shales are moderate to good source rocks, the mudstones are poor to good source rocks and the coals are very good source rocks (Peters and Cassa, 1994; Tissot and Welte, 1984). Hydrogen Index values also range from 41 to 327 mg HC/g TOC suggesting generally that the sediments are fair to good source rocks with potential for oil and gas (Peters and Cassa, 1994; Tissot and Welte, 1984).
4.2 Type of Organic Matter

Characterisation of organic matter in source rocks can be accomplished through a number of parameters from Rock-eval Pyrolysis measurements and their derivatives. The amount of hydrogen in kerogen is the most important factor with respect to the capacity of source rocks to generate petroleum (Hunt, 1996). Hydrogen-rich organic matter commonly generates more oil than hydrogen-poor organic matter (Demaison and Moore, 1980). Hydrogen index is a measure of hydrogen richness in kerogen and has a direct relationship with elemental hydrogen to carbon ratios. The index is used to define the type of kerogen and approximate level of maturation (Tissot et al., 1974). Van Krevelen diagram of Hydrogen Index (HI) against Oxygen (OI) for the sediments of Mamu Formation (Figure 4) shows that the organic matter contained in the sediments are predominantly type II/III and type III kerogen with subordinate type IV kerogen which are capable of generating oil and gas. Specifically, the coal samples within the sediments contained type II/III organic matter predominantly with only one sample giving type III kerogen (Figure 4). This view is also supported by the fact that HI values of the coal samples range from 167-327 mg/g with most values falling between 200 and 300 mg/g (Table 2, Peters and Cassa, 1994). The shale samples are characterised predominantly by type III kerogen with contribution from type II/III kerogen (HI value range of 50-288 mg/g, with a value above 200 mg/g). The mudstone samples also contained predominantly type III kerogen but with contributions from type II/III and type IV kerogen respectively (Figure 4, Table 2). The shales and mudstones contained predominantly type III kerogen while the coals are predominated by type II/III kerogen.

![Figure 4: Plot of Hydrogen Index (HI) against Oxygen Index (OI) for Mamu Formation sediments.](image)

Furthermore, the plot of HI against Tmax (Figure 5) indicates type II/III and III kerogen with subordinate type IV organic matter in immature window for the sediments. Specifically, the coal samples contained predominantly type II/III kerogen, while the shales and mudstones contained predominantly type III kerogen respectively. All the samples are within the immature window. Also, there are contributions of type III kerogen to the coals, type II/III to shales and type II/III & IV...
kerogen to the mudstones. In addition, the plot of HI against calculated vitrinite reflectance (% Ro) shown in Figure 6 suggests type II/III and type III kerogen as the main organic matter with subordinate type IV kerogen in immature to oil window. The coal samples are essentially composed of type II/III kerogen with all samples in immature window except one. The shale samples predominantly contained type III organic matter with all the samples within the oil window except two samples in immature window. Also, the mudstone samples are composed of type III kerogen with about 62 % of the samples in the oil window and the remaining samples in the immature window. Cross plot of S2 against TOC is an important tool for comparing petroleum generative potential of source rocks (Langford and Blanc-Valleron, 1990; Peters, 1986). Plot of S2 against TOC (Figure 7) for the sediments indicates that they are capable of generating oil and gas. All the coal samples except one and a sample each from the shales and mudstones are capable of generating oil and gas, while a sample each among the coals and mudstones and two shale samples are gas prone, and all the remaining samples are either organically lean or inert.

4.3 Thermal Maturity of the Organic Matter
Thermal maturity (Tmax) provides an indication of source rock maturity but it is influenced by source rock organic matter type and the presence of excess free hydrocarbons together with other factors like mineral matter content, depth of burial and age (Tissot and Welte, 1984). Peters (1986) suggested that a thermal maturity equivalent to a vitrinite reflectance of 0.6 % (Tmax 435°C) of rocks with HI >300mg HC/gTOC produces oil, PI and Tmax values less than about 0.1 and 435°C respectively indicate immature organic matter while PI and Tmax ranges of 0.1 to 0.4 and 435 to 450°C respectively indicate organic matter from early to the peak of maturation respectively. Tmax more than 450°C also indicate that the organic matter is over mature. The degree of thermal evolution of the sedimentary organic matter for the samples deduced from Rock-eval data such as Tmax, Production Index (PI) and calculated vitrinite reflectance (% Ro) shows that the maturity of shows that the maturity of the sediments ranges from immaturity to early maturity. Plots of PI versus Tmax (Figure 8) further shows that sediments are predominantly immature with respect to hydrocarbon generation and all the samples are at low level conversion except a shale sample which is marginally within the intensive generation phase of oil window. Though there is a mudstone sample above low level conversion but it is also within the immature window in the stained or contaminated phase.

Figure 5: Plot of Hydrogen Index (HI) against Tmax for the sediments belonging to Mamu Formation.
Figure 6: Plot of Hydrogen index against Calculated vitrinite reflectance for Mamu Formation sediments.
Figure 7: Plot of S2 against TOC for the sediments of Mamu Formation.

5.0 Conclusions
Sediments belonging to Mamu Formation exposed at Enugu, Okaba, Owukpa and Ezimo are composed of sandstones, siltstones, sandy shales, mudstones, shales and coals. The sandstones are white coloured and fine to medium grained while the siltstones are fine grained, gritty and white to grey in colour. The sandstones are fine to medium grained, white to yellowish brown in colour and sometimes crossbedded or laminated. The siltstones are fine grained and white to light brown in colour. The sandy shales are fine to medium grained, grey in colour and fissile. The mudstones are fine grained, grey to black in colour with the presence of fine whitish sandstone or siltstone giving it a stripped texture. The mudstones are sometimes concretionary with characteristic reddish brown colouration. The shales are fine grained, fissile and dark grey in colour. The coals are black coloured, fine grained, sometimes with lamination and may be fractured.
Figure 8: Plot of Production Index against Tmax for the sediments of Mamu Formation sediments.

The Total organic carbon (TOC) of the sediments ranges from 0.07 to 67.68 wt. % with 45.56 to 67.68 wt. % in the coals, 0.07 to 5.65 wt. % in the shales, and 0.12 to 4.46 wt. % in the mudstones, suggesting that they are potential source rocks for hydrocarbons. Generative Potential and Hydrogen Index indicate that the shales are good source rocks, the mudstones are poor to good source rocks, while the coals are very good source rocks, with all the sediments generally having potential for oil and gas generation. The organic matter types in the sediments are predominantly type II/III and type III kerogen with subordinate type IV kerogen which are oil and gas prone. The coals are dominated by oil and gas prone type II/III kerogen, while the shales and mudstones are dominated by gas prone type III kerogen. Thermal maturity derived from Rock-eval data indicates that the sediments are predominantly immature with respect to hydrocarbon generation and are generally at low level conversion. It is hereby concluded that the organic matter ranges from immaturity to the peak of maturity (late diagenetic stage to peak catagenetic stages of hydrocarbon formation). The Tmax and PI ranges of 412 to 432 and 0.02 to 0.08 respectively for the sediments indicate immaturity. However, % Ro range of 0.26 to 0.62 hydrocarbon potentials of the oil and gas prone coals and associated gas prone shales and mudstones in the Mamu Formation of Anambra basin can only be attained at appropriate maturity.

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