OPTICAL (VISUAL) KEROGEN ASSESSMENT OF ENUGU SHALE, ANAMBRA BASIN, SOUTHEASTERN NIGERIA: IMPLICATIONS FOR SOURCE ROCK POTENTIAL AND THERMAL MATURATION

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ABSTRACT

Palynological analysis was carried out on Ten (10) samples from outcrops of the Campanian Enugu Formation, a component lithostratigraphic unit of the Anambra Basin, using the acid maceration techniques for recovering acid-insoluble organic-walled microfossils. Two main lithological units were encountered, which include: carbonaceous shale and siltstone. Result from kerogen laboratory examination reveals two (2) main groups of palynofacies association namely; palynofacies (A and B), based on the change in particulate organic matter constituents of organic residue extract. Palynofacies A is characterized by abundant opaques debris with common terrestrial phytoclasts, which occupy the southwestern and northwestern parts of the studied area, whereas palynofacies B dominates in the northeastern part, characterised by abundant phytoclasts followed by frequent opaques debris. Kerogen type III with gas-prone material is suggested for both palynofacies. The examined exine of spore/pollen grain are pale yellow–yellow, with Thermal Alteration Index TAI of 1+ to 2- and Vitrinite Reflectance (R o) of 0.3 % - 0.4 % in palynofacies A, and yellow – yellow brown, with Thermal Alteration Index TAI of 2- to 2, and Vitrinite Reflectance (R o) of 0.3% - 0.5% in palynofacies B. These revealed source rock that is thermally immature to slightly mature but has potential to generate mainly gas. The kerogen data generated using transmitted light microscopy correlated well with geochemical data obtained using rock-eval pyrolysis method, and this shows the method a reliable tool for assessing petroleum potential in any given sedimentary basins.

KEYWORDS: Kerogen, Palynofacies, Anambra Basin, Cretaceous, Spore/ Pollen, Exine

1. INTRODUCTION

The Anambra Basin overlies Benue Trough in the southwestern and northern parts (Fig. 1 and Fig. 2). The basin is one of the seven sedimentary domains of Nigeria (Nwajide, 2006). It is a broad synclinal structure consisting of more than 5,000 ft thick of Upper Cretaceous sediments, representing the third phase of marine sedimentation in southern Nigeria (Ladipo, 1988; Akande and Erdtman, 1998; Ojo et al., 2009; Chiadikobi and Chiaghanam, 2018), and presents an economically viable hydrocarbon deposits (Akaegbobi, 2005).

The basin rests on the Awgu Formation in the southern Benue Trough, demonstrably over an angular unconformity. Its total area is thus possibly in excess of 14,000 km² (Nwajide, 2006). The stratigraphic package (Fig. 3) of the Anambra Basin is theorized to have originated as a result of the depression of the area around the southern Benue Trough contemporaneous with folding of the latter in the Santonian. The basin became filled with two lithological groups- the Nkporo Group and the Coal Measures respectively, following one major transgression and one major regression (Nwajide, 2006). The Nkporo Group

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forms the basal part of the Anambra Basin and consists of Nkporo Formation, Enugu Shale, Owelli/Otobi Sandstone, and Afikpo Sandstone. This lithological group reflects a funnel-shaped shallow marine shelf setting that graded into channeled low-energy marshes. The Coal Measure Group forms the upper part of the Anambra Basin and consists of the Mamu Formation overlain by the Ajali Formation and above by the Nsukka Formations. These accumulated during this epoch of overall regression of the Nkporo Group with associated progradation.

The petroleum geochemistry, sedimentology, biostratigraphy and depositional environments of various formations in the Anambra Basin have been discussed by various authors (Petters, 1978; Agagu et al., 1985; Ehinola et al., 2005; Akaegbobi, 2005; Ojo et al., 2009; Ekweozor, 2006). Some of them have showed the usefulness of organic geochemical methods in assessing generative potential and characteristics of source rocks in the Nkporo / Enugu Shales (Avbovbo and Ayoola, 1981; Whiteman, 1982; Ekweozor, 1982; Obaje et al., 1999; Nwajide, 2006; Anozie et al., 2014; Chiadikobi and Chiaghanam, 2018). Anozie et al. (2014) and Chiadikobi and Chiaghanam (2018) however concluded that organic facies of Nkporo / Enugu Shales are dominated by terrestrially-land derived organic residue that is largely immature associated with type III kerogen, and have potential to generate mainly gas.
Fig. 2: Regional stratigraphy of southeastern Nigeria showing the petroleum system elements of the Anambra Basin (modified after Nwajide, 2006)

These studies were based on geochemical methods using rock-eval pyrolysis. On the other hand, very few workers have demonstrated the use of transmitted light microscopy in assessing the generative potential and quality of source rocks in the Enugu Formation (Chiaghanam et al., 2013). In this study, attempt was made to compare kerogen results obtained, using visual (optical) transmitted light microscopic technique, with organic geochemical parameters obtained through rock-eval pyrolysis methods, in order to ascertain their correlatability.

This paper attempts to:
1. Delineate lithological successions of the Enugu Formation,
2. Determine palynofacies association and kerogen type,
3. Examine spores/pollen exine color, estimate Thermal Alteration Index (TAI), Vitrinite Reflectance (Ro%), and thermal maturation status and,
4. Assess petroleum potential and source rock quality of the Enugu Shale.

2. MATERIALS AND METHODS
As shown in Figure 2, samples were collected from mostly the exposures along the road cuts in the northern 20 km of the Enugu – Port Harcourt express way, i.e. from Ozalla junction to the NNPC Mega-station. Some more samples were picked from locations to the northwest of the study area. Special care was taken to ensure that only fresh samples were collected.

Ten (10) kerogen slides were prepared from shale samples, one for each sample, using standard conventional method of acid demineralization for recovering the acid-insoluble organic residue extract. Each kerogen-containing residue slide was carefully
studied using transmitted light microscopy at x10 and x40 magnifications, in order to:

i) carry out a qualitative as well as a quantitative analysis of dispersed particulate organic matter (POM)
ii) determine the palynofacies association and kerogen types
iii) examine spore/pollen exine color
iv) estimate Thermal Alteration Index (TAI), Vitrinite Reflectance (R₀ %), as well as the degree of organic thermal maturation

Each slide was scanned and counted for its POM contents. The first 200 particles were considered in terms of rare (<5%), common (5 – 15%), frequent (16 – 35%), and abundant (>35 %), (cf. Ibrahim et al., 1997; Zobba, 2007; Chiaghanam et al., 2013; Ikegwuonu et al., 2019).

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3. RESULTS

Table 1 shows the percentage frequency distribution of the total POM present in the samples. From the illustration given in the histogram (Fig. 4), it is evident that POM, such as opaque debris, overwhelmingly dominant especially in samples EN/L3/S4, EN/L5/S6 and EN/L7/S8, with up to 60% of the total counts (see Table 1) (Fig. 4). Other POM, such as phytoclasts are frequent, with highest percentage count of 45% in sample EN/L6/S7 followed by palynomorphs, with 25% frequency count in the same sample. Amorphous Organic Matter (AOM) is sparsely distributed, but common to rare in samples OZ/L1/S1, OZ/L2/S3, EN/L3/S4 and EN/L4/S5, with the corresponding frequency percentages of 5%, 3%, 5% and 2% respectively (see Table 1).

In general, all the samples from Locations 1, 2b, 3 and 4, documented the entire (4) main category of POM, including phytoclasts, opaque debris, AOM and palynomorphs. Meanwhile, samples from Locations 2a, 5, 6, 7, 8, and 9 recorded only the land-derived plant particles, the phytoclasts, opaque debris and palynomorphs, with no AOM from marine source (Fig. 5).

| SAMPLE NO. | PHYTOCLAST | OPAQUES | AOM | PALYNOMORPHS |
|------------|------------|---------|-----|--------------|
| OZ/L1/S1   | 40%        | 50%     | 5%  | 5%           |
| OZ/L2/S2   | 40%        | 55%     | 0%  | 5%           |
| OZ/L2/S3   | 35%        | 55%     | 3%  | 7%           |
| EN/L3/S4   | 25%        | 60%     | 5%  | 10%          |
| EN/L4/S5   | 25%        | 55%     | 2%  | 18%          |
| EN/L5/S6   | 30%        | 60%     | 0%  | 10%          |
| EN/L6/S7   | 45%        | 30%     | 0%  | 25%          |
| EN/L7/S8   | 20%        | 60%     | 0%  | 20%          |
| EN/L8/S9   | 40%        | 50%     | 0%  | 10%          |
| EN/L9/S10  | 30%        | 55%     | 0%  | 15%          |

Fig. 4: Histogram of % frequency distribution of the total Particulate Organic Matter (POM) present in the samples.
4. INTERPRETATION AND DISCUSSION

4.1 Palynofacies association
Two palynofacies associations: A and B, were recognised, based on the significant change in their constituent organic matter contents (Table 2).

**Palynofacies A:** This is characterized by an overwhelming abundance (50 – 60 %) of opaque debris followed by frequent phytoclasts. AOM and palynomorphs were also recorded but in low amounts (see Table 2). The opaque debris consists mostly of dark equant fine particles of land-derived organic material. Phytoclasts are both structured and unstructured plant particles. The structured phytoclasts include wood remains – tracheids, leaf cuticles, plant tissues – parenchyma and collenchyma, and cortex. Others are fungal remains such as hydrae and fruiting bodies. The structureless phytoclasts include degraded woods and plant resins. The AOM consists mostly of transparent fluffy organic matter of marine origin while palynomorphs include fern spores, pollen, algae – unicellular and multicellular, dinoflagellates, and foram test linings. Kerogen samples from the Locations 1, 2, 3, 4, 5, 7, 8 and 9, yielded similar palynofacies association, and therefore are grouped under this palynofacies association.

**Palynofacies B:** This is characterized by abundant phytoclasts and frequent opaque debris, followed by palynomorphs and then AOM. (see Table 2). The phytoclasts constituted mostly plant tissues and xylems while opaque debris is mainly of small equant fine particles. Palynomorph constitutes mostly of fresh water fern spores and pollen grains of mangrove affinity. Fungal spores and other organic matters from marine origin such as, dinoflagellates, foram test lining, acritarchs, fluffy substance, and structureless dark debris, are rare. Only sample EN/L6/S7, from Location 6, contains this palynofacies association, and is grouped under this palynofacies zone.

4.2 Kerogen type and quality
Kerogen residue extracts from all the samples are dominated by POM typical of terrestrial origin such as opaque debris and structured phytoclasts. Following the dominance of land-derived organic matter over the AOM of marine origin in all the samples, a gas-prone material with type III kerogen is proposed for the entire study area (Table 2). This is evidenced by the presence of a large amount of structured woody plant material, especially xylem, and leaf cuticles, together with an abundance of carbonized wood and opaque particles.

4.3 Spores / pollen exine color
The color of fossil spore/pollen exine is a useful measure of organic material maturity or carbonization (Jiang et al., 2016). The color of palynomorphs relates directly to petroleum generation potential, thermal alteration index (TAI), and vitrinite reflectance (R₀). In correlating and comparing fossil exine color change using Pearson (1982) standard color chart, pale yellow and yellow exine fall in the immature phase while orange and brown tissues are in the liquid petroleum and wet gas range. Dark brown ones are in the dry gas and
condensate range (Fig. 6). This information allows predictions for the petroleum source rock quality. In this study, kerogen samples were examined to estimate the color change of fossil exine and compare them using the Pearson’s color chart. It is observed that almost all the samples, except the one from Location 6 (EN/L6/S7), yielded fossil color within the range of pale yellow – yellow. It however produced spores/pollen with yellow to yellow brown exine.

4.4 Organic thermal maturation, thermal alteration index, and vitrinite reflectance (R_o %)

Organic thermal maturity provides an indication of maximum paleo-temperature reached by source rock (Ogala, 2011). As the depth of burial increases, organic matter in the sediments experiences gradual change in composition due to temperature increase. As a result, the original nature of organic matter within the sediments is altered in response to the ongoing digenetic processes. This process tends to pose significant changes especially in the original color of palynomorphs. This change in color of palynomorphs tends to preserve the thermal record and burial history of sediments containing them, which has been found useful in the oil industry as an alternative tool in evaluating organic thermal maturity, and for estimating thermal alteration index and vitrinite reflectance.

Also from spore/pollen examination, it is observed that samples from the Locations 1, 2, 3, 4, 5, 7, 8, and 9, generally indicate palynomorph color of pale yellow – yellow, which falls within the early immature zone as made out from the Pearson’s color chart, and hence, are largely immature (Fig. 6). This corresponds with the thermal alteration index (TAI) value of 1+ to 2−, and vitrinite reflectance.

| SAMPLE NO | PALYNOFACIES ASSOCIATION | S/P COLOUR          | TAI          | VITRINITE REFLECTANCE (R_o %) | THERMAL MATURATION       | KEROGEN TYPE | SOURCE ROCK POTENTIAL |
|-----------|-------------------------|---------------------|--------------|-------------------------------|--------------------------|--------------|-----------------------|
| OZ/L1/S1  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| OZ/L2/S2  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| OZ/L2/S3  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L3/S4  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L4/S5  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L5/S6  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L6/S7  | Abundant phytoclast & frequent opaque | Yellow - yellow brown | 2− to 2     | 0.3 % - 0.5 %                | Immature - slightly mature | TYPE III     | Gas prone             |
| EN/L7/S8  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L8/S9  | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
| EN/L9/S10 | Abundant opaque & frequent phytoclast | Pale yellow - yellow | 1+ to 2−     | 0.3 % - 0.4 %                | Largely Immature         | TYPE III     | Gas prone             |
reflectance ($R_o$) 0.3% - 0.4%. Sample (EN/L6/S7) from Loc. 6, shows spore/pollen color of yellow – yellow brown, and falls within the late immature zone. This corresponds to the thermal alteration index value of 2- to 2 and vitrinite reflectance of 0.3% - 0.5%, (Table 2).

4.5 Petroleum source rock potential of Enugu Shale

Examination of dispersed particulate organic matter using transmitted light microscopy has become an important tool for assessing the petroleum potential of sedimentary basins (Batten, 1982). The major components of palynofacies and the exine colour of the palynomorphs preserved therein are widely utilized in the oil industry to aid the determination of organic maturity level and source rock potential for hydrocarbons. The integration of palynological and sedimentological data has equally played an important role in facilitating the identification of depositional environments (Batten, 1974). Differences in depositional environments control, to a great extent, the composition and type of organic particles in a given sedimentary basin, and also determine the hydrocarbon quality and types. Palynofacies dominated by land plant detritus tends to produce type III kerogen, and thus constitute good source rock for gas. On the other hand, Botryococcus and amorphous matter of marine/algal origin, if abundant in sediments, tend to produce kerogen types I and II, which are precursors for oil (Tissot and Welte, 1978).

The presence of different kinds of terrestrial fossils in the studied sections indicates deposition in continental to shallow marine water pro-deltaic settings. This suggestion is reinforced by the light grey nature of the shales, indicating low content of carbonaceous matter. The documented organic facies change reflects a shift in the paleoenvironmental conditions in response to sea level changes and sediment contribution to the basin. Palynofacies A, which contains large amounts of land-derived opaque debris, followed by phytoclasts, may have been deposited in a shallow marine setting proximal to active fluvial influx. This suggests kerogen type III with gas-prone material, though still largely immature, with palynomorph color of pale yellow – yellow. All the samples from Locations 1, 2, 3, 4, 5, 7, 8, and 9, at Ozalla, Independence Layout, New Heaven, and Trans Ekulu, fall within this palynofacies association (Fig. 6). Palynofacies B, on the other hand, contains higher amounts of phytoclasts with common opaques, reflecting a shallower depositional setting which the marine elements could not effectively reach. These suggest the same kerogen type III with gas-prone material, but are still organically immature to slightly mature, characterised by the observed palynomorph color of yellow – yellow brown. The sample from Location 6 (EN/L6/S7), collected at Emene River, falls within this palynofacies association (Fig. 6).

4.6 Comparison with previous geochemical studies in the formation

Several workers have demonstrated the usefulness of organic geochemical methods in assessing the generative potential and characteristics of source rocks in the Nkporo / Enugu Shales (Avbovbo and Ayoola, 1981; Whiteman, 1982; Ekweozor, 1982; Obaje et al, 1999; Nwajide, 2006; Anozie et al., 2014; Chiadikobi and Chiaghanam, 2018). However, very few workers have applied transmitted light microscopy in evaluating the generative potential and quality of source rocks in the formation (Chiaghanam et al., 2013). In this study, attempt was made to compare results obtained through (visual) transmitted light microscopy with the existing organic geochemical parameters obtained by various workers using rock-eval pyrolysis methods, so as the ascertain their correlatability (Fig. 7).

Conclusions /Recommendation

Kerogen assessment using visual (optical) transmitted light microscopic method has demonstrated high confidence level, as a viable tool, in assessing petroleum source rock potential and thermal maturity status in the study area. Kerogen data obtained through palynological laboratory analysis reveals two main palynofacies associations; palynofacies A and palynofacies B, based on significant difference in organic particle contents. Both palynofacies produced kerogen type III with (gas-prone material) following the predominance of land-derived plant particles of opaque debris and phytoclasts, with few marine elements. The changes in color of particulate organic matter (especially miospores), reveal color range of pale yellow – yellowish brown, indicating a type of organic matter content that is largely immature to slightly mature but with a potential to generate gas.
Fig. 6: Composite chart of the studied sequence showing litho-sections & sample locations, lithologic description, palynofacies and kerogen type, and thermal maturity.

| Age | Formation | Petroleum System Element | Ekweozor (1982) | (Obaje et al., 1999) | Anozie et al., 2014 | This Study |
|-----|-----------|--------------------------|----------------|----------------------|---------------------|------------|
|     |           |                          | TOC (wt%) | SOM (wt%) | Maturity Status | Kerogen Type | TOC (wt%) | V/R | Maturity Status | Kerogen Type | TOC (wt%) | Maturity Status | Kerogen Type |
| Danian | Nsukka | Cap Rock/ Regional Seal | 1.6 – 5.3 | 28.1 | Largely Immature | Type III (Gas) | 1.1 – 4.2 | 0.44 – 0.60 | Immature | Type III (Gas) | 1.56 – 4.41 | Immature to fairly mature | Type III (Gas) | 1- to 2- | 0.30 – 0.50 | Immature to slightly immature | Type III (Gas) |
| Maatrichitan | Ajali | Reservoir | | | | | | | | | | | | | |
| | Mamu | Source Rock | | | | | | | | | | | | | |
| Companian | Nkporo/ Enugu | Source Rock | 1.6 – 5.3 | 28.1 | Largely Immature | Type III (Gas) | 1.1 – 4.2 | 0.44 – 0.60 | Immature | Type III (Gas) | 1.56 – 4.41 | Immature to fairly mature | Type III (Gas) | 1- to 2- | 0.30 – 0.50 | Immature to slightly immature | Type III (Gas) |

Fig. 7: Correlation/comparison chart showing the elements of possible hydrocarbon system of the Anambra Basin, and source rock potential and maturity status of the Nkporo/Enugu Shale according to the given authors.
Despite its subjectivity, transmitted light microscopic study of dispersed organic matter is a relatively cheap, quick and, surprisingly accurate technique for evaluating maturation levels and recognizing hydrocarbon source rock facies. It is, therefore, an invaluable tool for assessing petroleum potential in sedimentary basins. In order to ensure reliable conclusions, it is recommended that data of such analyses and their implications should be correlated and compared routinely with the results from vitrinite reflectance, fluorescence and geochemical studies on the same stratigraphic sections.

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