Palynology, Source Rock Potential and Thermal Maturity of Eocene Nanka Formation (Ameki Group) In Anambra Basin: An Investigation of Agulu Lake, South-Eastern Nigeria.

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Abstract: Palynological and hydrocarbon potential of Eocene Nanka Formation was investigated from subsurface shale sample recovered between the depth 17 to 28 meters at Agulu Lake south eastern Nigeria. The Rock-eval pyrolysis results recorded shows that Total organic carbon (0.40), S_1 (0.03) and S_2 (0.23) of the analyzed sample has a petroleum potential that is poor in quality. Production Index (0.10) and Tmax (412) records indicates that the stage of thermal maturity for its oil is mature. Hydrocarbon Index (58) and Oxygen Index (101) results suggest a Type IV Kerogen. The kerogen investigation indicates that particulate organic matter, spore and pollen colouration and vitrinite reflectance results obtained from the simple is at an immature thermal oil maturity stage. The age-diagnostic index palynomorphs species recovered assigned a Middle-Eocene for the studied area. The results suggests that Agulu lake, which is mid-Eocene has a hydrocarbon record of type IV kerogen, immature stage of thermal maturity, poor source bed, but has organic matter that may generate oil.

Keywords: Nanka Formation, Anambra Basin, Agulu Lake, Rock-eval Pyrolysis, Kerogen and Palynomorphs

I. Introduction

The sedimentology, sequence stratigraphy and hydrocarbon potentials of Eocene (Nanka Formation) sedimentary fill of the Anambra Basin, Southeastern Nigeria, have been studied by various researchers (Nwajide, 1979, 1980; Nwajide and Reijers, 1996; Umeji, 2003; Nwajide, 2006; Reyment, 1965; Chiaghanam 2008; Chiaghanam et al 2014a, 2014b). Nwajide (1979 and 1980) Demarcated Nanka Formation as the loose sand facies of the Ameki Group, which noted it to be very succeptible to gully erosion. Nwajide and Reijers (1996) and Umeji (2003) studied the type locality and other gully sections, road cut exposures (reference localities) of Naka Formation. Nwajide (2006) used the section exposed near Rhema maintain Cathedral at Umunya to demonstrate Walter's law, clastic sedimetary structures, reservior properties, sequence stratigraphic principles and variability environment diagnosis. Reyment (1965) placed the age of the Nanka Formation at the Earliest Eocene (Tpresian). Chiaghanam (2008) used lithofacies and paleoenvironmental approach to study the Eocene sediments of Umunya section of Nanka Formation. Chiaghanam et al (2014a) applied geochemical parameters in describing the source rock and thermal maturity of Nanka Formation using Ogbunike quarry site reference locality as a case study for basin wide prediction. Chiaghanam et al (2014b) studied the sedimentology and sequence stratigraphy of Ogbunike quarry site reference locality as a case study for basin wide prediction.

This study attempts to combine the palynological and Geo-chemical informations obtained from the subsurface section of Agulu Lake to interprete the paleoenvironments, age determination and hydrocarbon potentials of Agulu Lake in Eocene Nanka Formation of Anambra Basin, Southeastern Nigeria. It is also important to note that the renewed search for hydrocarbon in the inland basins has gone beyond seismic acquisition to stratigraphic and facies studies. Most of the world's largest oil blocks are located within the siliciclastic sedimentary environment and their diversity and stratigraphic heterogeneity to the diverse and different depositional environments in which they were deposited (Magoon and Dow, 1994; Nwachukwu et al, 2011; Chiaghanam et al. 2013 and 2014a).

Geological Setting

Anambra Basin is located in the South Eastern part of Nigeria. The Basin is bounded to the North by Bida Basin and Northern Nigeria Massif, to the East by Benue Trough, to the west by West Africa Massif and to the South by the Niger Delta Complex (Fig 1).

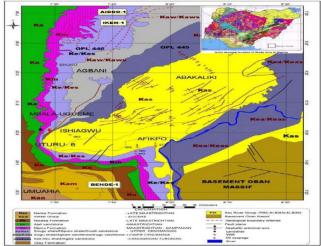


Fig. 1: Geologic Map of the Anambra Basin (adopted from Babatunde O.L. 2010)

The basin is a Cretaceous Basin having almost a roughly triangular shape with a total sediments thickness of about 9km, covers an area of about 40,000 sq.km. The Basin is characterized by enormous lithological heterogeneity in both lateral and vertical extensions derived from a range of paleo-environmental settings (Akaegbobi, 2005). Anambra Basin is a structural (synclinal) depression and one of the intracratonic basins in Nigeria whose origin is related to the separation of Africa from South America and the opening of South Atlantic Ocean (Ofoegbu, 1982). The basin developed due to the Santonian tectonic event which greatly affected Benue Trough, terminating sedimentation in the Abakaliki Basin but before then, sedimentation in Southern Nigeria which Started during Early Cretaceous was facilitated by the break-up of the Africa and South America continent resulting in the formation of Benue Trough (Burke et al 1972, Benkelil, 1989). Sedimentation in the trough was controlled by three major tectonic phases which resulted in the three successive depocentres (Murat, 1972, Oboh-Ikwunobe et al, 2005). The first phase (Albian - Santonian) was the period when Asu River Group, Eze-Aku and Awgu Formations within the Abakiliki - Benue Trough were deposited. The second phase (Campanian - Eocene) was characterized by compressive movements along NE-SW axis which resulted in the folding and uplift of the trough into an anticlinorium. This forced the Anambra Platform to subside and the depocenter to shift south-westwards to the newly formed Anambra Basin and the Afikpo Syncline on the other side of the anticlinorium in the southeast. The deposition of the Nkporo Group, Mamu Formation, Ajali Sandstone, Nsukka Formation, Imo Formation and the Ameki Group then followed. Imo Formation and Ameki Group were later deposited. Third phase commenced towards the end of Eocene and this saw the structural inversion of the Abakaliki region further shifting the depocenter down dip to the South to form the Niger Delta Basin (Obi et al, 2001, Nwachukwu et al 2011). Table 1 below shows the lithostragraphic framework of Anambra Basin.

| AGE | | ABAKALIKI-ANAMBRA BASIN AF | IKPO BASIN |
|----------------|---------------|---------------------------------------|------------------------------------|
| 30 my | Oligocene | Ogwashi-Asaba Formation | Ogwashi-Asaba Formation |
| 54.9 <u>my</u> | Eocene | Ameki/NankaFormation/Nsugbe Sandstone | Ameki Formation |
| 60 my | Paleocene | Imo Formation Nsukka Formation | Imo Formation Nsukka Formation |
| 73 my | Maastrichtian | Ajalli Sandstone Mamu Formation | Ajalli Sandstone Mamu Formation |
| 83 my | Campanian | Nkporo/ Owelli Formation/Enugu Shale | kporo Shale/Afikpo Sandstone |

 Table 1: Lithostratigraphic Framework of Anambra Basin (after Nwajide, 1990)

| 87.5 my | Santonian | Non-deposition | $\sim\sim$ |
|---------|-----------------------------------|--|--|
| 88.5mj | Coniacian | Awgu Group (Agbani Sandstone/Awgu Shal | e) |
| 93 my | Turonian | Ezeaku Group) | Ezeaku Group (incl Amaseri Sandstone) |
| 100 my | Cenomanian-Albian | Asu River Group | Asu River Group |
| | Aptian Baremian Hauterivian | Unnamed Units | 1 |
| | Precambrian | BasementComplex | |

The lithology of Nanka Formation is overwhelming loose, flaser-bedded, fine to medium sand with a few mudrock breaks. The sand consists of subrounded to subangular grains and has an average of 5% clay content which makes it texturally submature it is however compositionally highly mature on account of the absence of feldspars and the dominance of the ultrastable heavy mineral suite- zircon, tourmaline and rutile (Nwajide, 2013).

The studied area, which is a lacustrine system, is located at lat 6° 08' 02. 41" N and log 7° 02' 06.51" E. Fig 2

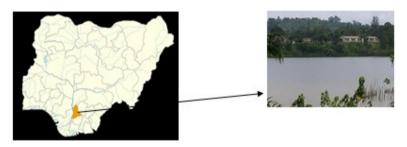


Fig. 2: Map of Nigeria with the picture of the Aerial View of Agulu Lake (www.wikipedia.com)

It has been observed that many lake sediments contain abundant fine organic matter that may act after burial as a source material for petroleum (Katz, 1990). Agulu Lake can be regarded as an under filled lake due to its persistently closed hydrology, characteristic chemicals stratification, highly contrasting lithologies and common association with evaporate deposits. Bohacs et al. (2000) and Boggs (2006) due to high sedimentation rates in lakes and the fact that Agulu Lake is essentially closed system with respect to sediment transport, they are regarded to face an ephemeral features. The filling process as observed in Agulu Lake shows a coarsening upward succession of the lake facies. Ideal coarsening-upward successions of lake sediments probably merely occur, except perhaps in some small lakes, of which Agulu Lake can be regarded as one, (Picard and High, 1981).

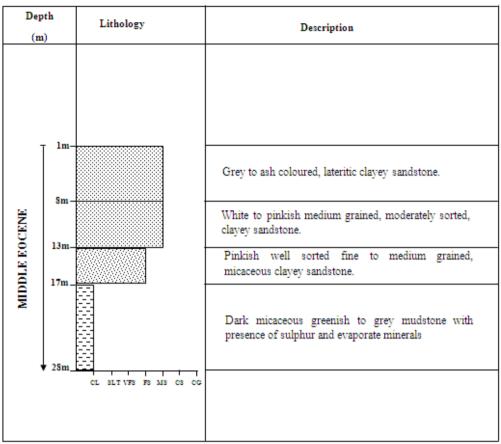


Fig. 2b shows the sub-lithologic log and its description of Agulu Lake.

II. Materials And Methods-

The location of the subsurface position was carried out using global positioning system application. The rock eval pyrolysis analysis of the shale sample recovered from the ditch cuttings at the depth of 17 to 28m below sea level was done at Gatemme Geochem Laboratory in Port-Harcourt, Nigeria. The samples were heated in an inert atmosphere to 550° c using special temperature programme, which produced S₁, S₂ and S₃ peaks. The first peak (S_1) indicates hydrocarbon already present in the sample which are striped at the temperature of 300°c. Second peak (S_2) indicates hydrocarbon that was generated due to thermal creaking of kerogen within the temperature range of 300-550°c. Third peak (S3) indicates CO₂ that are generated during the thermal cracking of kerogen. The temperature at maximum (Tmax) was recorded at S_2 peak by the instrument. Total organic carbon (Toc) and soluble organic matter (SOM) of the shale were also recorded; other important parameters such as Production Index (PI), Hydrogen Index (HI) and Oxygen Index (OI) were calculated and recorded. The determination of total organic carbon was done using the Walkey Black wet oxidation method. This assessment served as a preliminary screening for further detailed rock eval analysis. Kerogen investigation was also carried out on the sample to confirm results obtained from rock eval pyrolysis. During the analysis, slide from the units were examined using the transmitted light microscopy at x10 and 40 magnifications in order to make a qualitative as well as quantitative analysis of the Particulate Organic Matter (POM), determination of spore/pollen colouration and assessment of Thermal Alteration Index (TAI), Vitrinite reflection (Ro%) and Organic thermal maturation. The slides was counted for its (POM) content, in which the first 200 particles were counted in terms of abundant (>35%), frequent (15-35%), common (5-15%) and rare (less than 5%), (Ibrahim et al, 1997). The sample was also analyzed for palynomorphs using the maceration techniques. The sample was digested for 30 minutes in 37% hydrochloric acid to remove the traces of carbonate and 72 hours in 48% hydrofluoric acid to remove silicate. The extract were sieve- washed with water through 10 microns nylon mesh, oxidized for 30 minutes in 70% HN03 and 5 minutes in Schulze solution to render the fossils translucent for transmitted light microscopy. The oxidized residues were rinsed in 2% KOH solution to neutralize the acid. Swirling treatment was undertaken in order to get rid of the resistant coarse inorganic mineral particles. The residues were stained with Safranin-0 to increase the contrast for study and photography. Aliquots were dispersed with polyvinyl alcohol, dried on cover -slips and mounted in petro-poxy resin. Light photomicrographs were taken with a Leica Galen III microscope

III. Results And Interpretations

The level of maturity of the sample recovered will be dependent on depth, temperature and time of burial. The result of rock eval pyrolysis indicates as follows.

- 1. Total Organic Carbon (TOC, wt. %)= 0.40
- 2. $S_1 (mg HC/g rock) = 0.03$
- 3. $S_2 (mg HC/g rock) = 0.23$
- 4. $S_3 (mg HC/g rock) = 0.40$
- 5. T_{max} (°C) = 412
- 6. Hydrogen index [HI =(S_2 /Toc) x 100,mg HC/gToc] = 58
- 7. Oxygen index $[OI = (S_3/Toc) \times 100, mg CO_2/gToc] = 101$
- 8. $S_2/S_3 = 0.6$
- 9. $S_1/Toc \ge 100 = 8$
- 10. Production or productivity index $[PI = S_1/(S_1+S_2)] = 0.10$

The Interpretation of the above results of subsurface shale sample recovered between 17 to 28 m depth of Agulu lake was based on Peters and Cassa, 1994 Geochemical parameters which described (a) Petroleum potential (quality) of an immature source rock (b) Kerogen Type (quantity) and the character of expelled products (c) level of thermal maturation. See table 2.

Table 2.1 Geochemical Parameters Describingthe Petroleum Potential (Quantity)

| | Organic Matter | | | | | | | |
|-----------|----------------|----------|------------------|-----------|-------------------|--------------|--|--|
| Petroleum | TOC | Rock-Eva | I Pyrolysis | Bit | umen ^c | Hydrocarbons | | |
| Potential | (wt. %) | S1ª | S ₂ b | (wt. %) | (ppm) | (ppm) | | |
| Poor | 0-0.5 | 0-0.5 | 0-2.5 | 0-0.05 | 0-500 | 0-300 | | |
| Fair | 0.5-1 | 0.5-1 | 2.5-5 | 0.05-0.10 | 500-1000 | 300-600 | | |
| Good | 1–2 | 1–2 | 5-10 | 0.10-0.20 | 1000-2000 | 600-1200 | | |
| Very Good | 2-4 | 2-4 | 10-20 | 0.20-0.40 | 2000-4000 | 1200-2400 | | |
| Excellent | >4 | >4 | >20 | >0.40 | >4000 | >2400 | | |

amg HC/g dry rock distilled by pyrolysis.

^bmg HC/g dry rock cracked from kerogen by pyrolysis.

•Evaporation of the solvent used to extract bitumen from a source rock or oil from a reservoir rock causes loss of the volatile hydrocarbons below about nC15-. Thus, most extracts are described as *C15+, hydrocarbons. "Lighter hydrocarbons can be at least partially retained by avoiding complete evaporation of the solvent (e.g., C10+).

| Table 2.2 Geochemical Parameters Describing Kerogen | Type (Quality) and the character of Expelled Products |
|---|---|
|---|---|

| | н | | | Main Expelled Product |
|--------------|---------------|--------------------------------|------------|-----------------------|
| Kerogen Type | (mg HC/g TOC) | S ₂ /S ₃ | Atomic H/C | at Peak Maturity |
| | >600 | >15 | >1.5 | Oil |
| 11 | 300-600 | 10-15 | 1.2-1.5 | Oil |
| 11/111p | 200-300 | 5-10 | 1.0-1.2 | Mixed oil and gas |
| 111 | 50-200 | 1–5 | 0.7-1.0 | Gas |
| IV | <50 | <1 | <0.7 | None |

aBased on a thermally immature source rock. Ranges are approximate.

bType II/III designates kerogens with compositions between type II and III pathways (e.g., Figure 5.1) that show intermediate HI (see Figures 5.4–5.11).

| Table 2.3 | Geochemical | Parameters | Describing | Level of | Thermal Maturation |
|------------|-------------|--------------|------------|----------|--------------------|
| 1 abic 2.5 | Ocochemical | I arameters. | Describing | | |

| | Maturation | | | Generation | | |
|--------------------------------------|-----------------------|--------------------------|---------|------------------------------|------------------------|---|
| Stage of Thermal Maturity for Oil | R _o (%) | T _{max} (°C) | TAIa | Bitumen/ TOC ^b | Bitumen (mg/g rock) | PI ^c [S ₁ /(S ₁ + S ₂)] |
| Immature | 0.2-0.6 | <435 | 1.5-2.6 | <0.05 | <50 | <0.10 |
| Mature | | | | | | |
| Early | 0.6-0.65 | 435-445 | 2.6-2.7 | 0.05-0.10 | 50-100 | 0.10-0.15 |
| Peak | 0.65-0.9 | 445-450 | 2.7-2.9 | 0.15-0.25 | 150-250 | 0.25-0.40 |
| Late | 0.9-1.35 | 450-470 | 2.9-3.3 | _ | _ | >0.40 |
| Postmature | >1.35 | >470 | >3.3 | _ | | _ |

aTAI, thermal alteration index.

Mature cil-prone source rocks with type I or II kerogen commonly show bitumen/TOC ratios in the range 0.05–0.25. Caution should be applied when interpreting extract yields from coals. For example, many gas-prone coals show high extract yields suggesting oil-prone character, but extract yield normalized to TOC is low (<30 mg HC/g TOC). Bitumen/TOC ratios over 0.25 can indicate contamination or migrated oil or can be artifacts caused by ratios of small, inaccurate numbers.

The total organic carbon which described the quantity of organic carbon in a rock sample was recorded 0.40 from the analysis, which shows that the petroleum potential (quantity) of the analyzed sample is poor. The S_1 which measures hydrocarbon shows as the amount of free hydrocarbon that can be volatilized out of the rock

without cracking the Kerogen, has a value of 0.03 recorded from the analyzed sample which also indicated that the sample has a petroleum potential (quality) that is poor.

The S_2 which measures the hydro carbon yield from cracking of kerogen (mg Hc/g rock) and heavy hydrocarbon and represents the existing potential of a rock to generate petroleum, has a value of 0.23 recorded from the analyzed sample, which also indicates that the sample has a petroleum potential (quality) this is poor.

The Tmax which measures thermal maturity and corresponds to the rock- eval pyrolysis oven temperature ($^{\circ}$ C) at maximum S₂ generation has a value of 412 recorded from the analyzed sample, which suggest that the analyzed shale sample is still at stage of thermal maturity of oil that is immature.

The hydrogen index which is proportional to the amount of hydrogen in the kerogen and thus indicates the potential of the rock to generate oil, has a value of 58 recorded from the analyzed sample which suggest a type IV kerogen, shows a very low atomic H/C (0.5-0.6) and low to high O/C (\leq 0.3). The kerogen is dominated by inertinite macerals that generate little or no hydrocarbons during maturation.

The production or productivity index is the gradually increases with depth for fine grained rocks as thermally labile components in the kerogen (S_2) are converted to free hydrocarbons (S_1) , has a value of 0.10 recorded from the analyzed shale sample, which suggest an immature stage of thermal maturity for oil.

The S_3 measurement is not as reliable as other Rock-Eval parameters, partially because of interference of carbonate minerals or kerogen oxidation resulting from pulverizing the sample (Peters and Cassa, 1994). Based on the above facts S_3 was not applied in the study.

The plot of Hydrogen Index (HI,mg Hc/gToc) against Oxygen Index (OI, mgCO₂/g Toc) which shows the organic matter quality, Indicates that the analized shale sample is a Type IV Kerogen, see fig 3. While the plot of production index (P1) against maturity (based on Tmax, °c) which shows the kerogen conversion maturity, Indicates an immature thermal maturity for the analyzed shale sample, see fig 4.

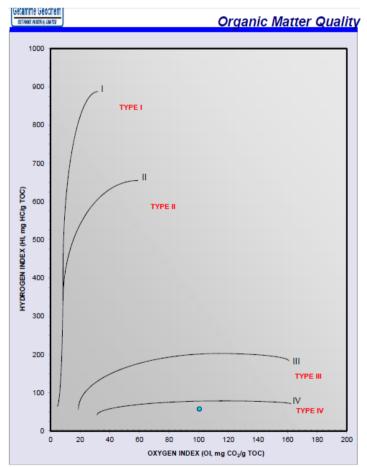


Fig. 3: The plot of Hydrgen Index (HI) against Oxygen Index (OI)

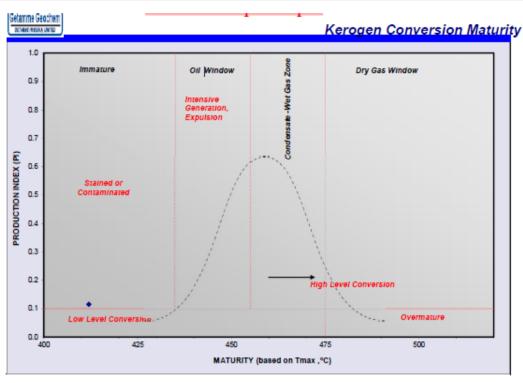


Fig. 4: The plot of Production Index (PI) against Maturity (Tmax, °C)

The kerogen investigation shows that the frequency distributions of the total particulate organic matter (POM) present in the analyzed sample are as follows

Phytoclast 3%

Amorphous organic matter (AOM) = 59.5% Opaques = 35.5%

Palynomorphs = 2%

Fig 5, shows the Histogram distribution of particulate organic matter (Pom) in the sample, while fig 6 is the micrographs of the kerogen slides showing the various (POM) of the examined sample.

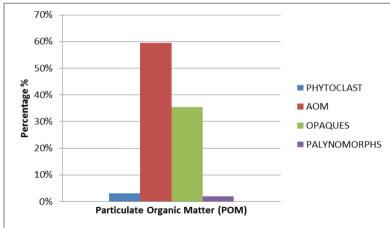


Fig. 5: Histogram % distribution of particulate organic matter (POM) in the kerogen sample.

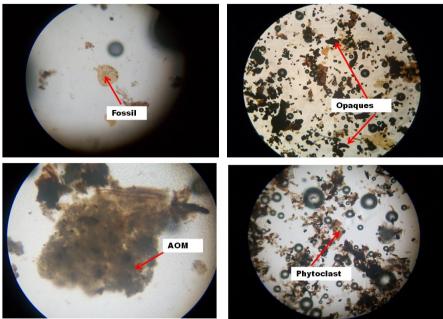


Fig 6: Micrographs of the Kerogen slides showing the various (POM) of the examined sample.

Based on Pearson 1984 colour chart, other results obtained from kerogen investigation were interpreted; the results were also confined from (Peters and Cassa 1994). The results are as follows

Spore/pollen colouration = yellow

Vitrinite reflection = 0.3 %Thermal Alteration Index (TAI) = -2

The spore/pollen colouration of yellow; thermal alteration index, which is a numerical scale based on thermal induced colour changes in spores and pollen with an analyzed value of -2 and vitrine reflection which increases during thermal maturation due to complex, irreversible aromatization reactions, with an analyzed value of 0.3%, all indicates that the sample from studied area has a thermal maturation stage that is immatured, fig 7 shows the (Pearson 1984) colour chart.

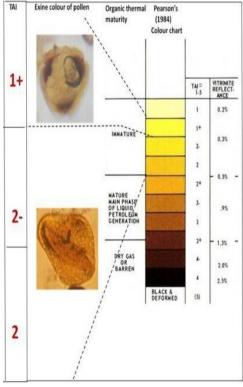


Fig. 7: Showing Thermal Alteration Index and Vitrinite Reflectance chart (Pearson, 1984)

The summary of the palynomorphs percentage (%) frequency distribution and paleoenvironmental inference of the analyzed shale sample from Agulu Lake indicates spores =38%, pollen =54%, marine species =8%, paleosalinity =Brackish water and paleoenvironments =marginal marine probably estuarine or lacustrine environment.

The palynomophs of environmental value encountered in the examined sample include Proxapertites cursus/operculatus, Spinizonocolpites echinatus and psilatricolporites crassus are pollen of brackish water palms inhabiting similar environment as that of mangrove swamp, (Umeji, 2002, Umeji and Nwajide ,2013) .The presence of species such as Cordosphaeridium sp. indicates restricted neritic to outer neritic depositional environment, (Van Mourik and Brinkhuis, 2001).

Sample from Agulu Lake was found to be laking definite age-diagnostic palynomorphs assemblage. However based on the following recovered age –diagnostic index palynomorphs species such as Retibrevitricolpites triangulates, Spinizonocolpites echinatus, Psilatricoplorites crassus, psilatricolporites operculatus and proxapertites cursus, a middle Eocene age was assigned to it (Van Hoekenklinberg, 1966; Germeraad et al, 1968). Table 3 and fig. 8, shows the occurrences and distribution of palynomorphs species in the analyzed sample from Agulu Lake.

| Sample No. | AGULU LAKE (SP 1) |
|-----------------------------------|----------------------|
| Palynomorphs species | |
| TERRESTRIAL SPECIES | |
| Spores | |
| Schizosporis parvus | - |
| Cyathidites minor | 2 |
| Leiotriletes adriennis | - |
| Leiotriletes maxoides | - |
| Verrucatosporites usmensis | - |
| Laevigatosporites ovatus | 6 |
| Cicatricosisporites dorogensis | - |
| Polypodiaceoisporites sp. | 2 |
| Pollen | |
| Psiltricolporites operculatus | 4 |
| Spinizonocolpites echinatus | 1 |
| Proxapertites operculatus | 3 |
| Proxapertites cursus | 1 |
| Retitricolporites irregulari | |
| Retistephanocolpites williamsi | - |
| Pachydermites diederixi | - |
| * | - |
| Ctenolophonidites costatus | - |
| Tricolpites hians | 1 |
| Striatopollis catatumbus | - |
| Monocolpites marginatus | - |
| Monoporites annulatus | - |
| Psilatricolporites sp. | 2 |
| Psiltricolporites crassus | 2 |
| Inaperturopollenites sp. | - |
| Retibrevitricolpites triangulatus | 2 |
| Psilatriporites rotundus | - |
| Echitriporites trianguliformis | - |
| Psilastephanocolporites sp. | - |
| Echitricolporites spinosus | - |
| Anacolocidites cf. luteoidies | - |
| Brevicolporites (P) molinae | - |
| Scabratriporites annulatus | - |
| Bombacidites sp. | - |
| MARINE SPECIES | |
| Dinoflagellate cysts | |
| Coronifera oceanica | - |
| Hafniasphaera septata | - |
| Achilleodinium biformoides | - |
| Diphyes colligerum | - |
| Homotryblium spp. | - |
| Cordosphaeridium inordes | 1 |
| Spiniferites hyperacanthus | 1 |

Table 3: The occurance and distribution of palynomorphs species in the analyzed samples

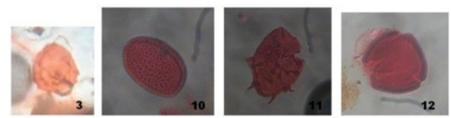


Fig. 8: Micrographs of some palynomorphs recovered from the analyzed sample.

Magnification (X 40)

- 3. Psilatricolporites operculatus
- **10.** *Proxapertites cursus*
- 11. Spinizonocolpites echinatus
- **12.** *Psilatricolporites crassus*

IV. Conclusion

The search for hydrocarbon in the inland Basin and observed fact that many lake sediments contain abundant organic matter which may act after burial as a source material, has necessitated this research work in Agulu Lake located within the Anambra Basin. The investigated shale unit and results obtained from Rock eval pyrolysis and kerogen analysis suggests a petroleum potential (quality) that is poor, Kerogen type IV and a thermal maturation stage of immature .The palynomorphs of environmental value encountered indicates a restricted depositional environment, while age – diagnostic index palynomorphs species assigned Middle – Eocene Age to Agulu Lake sediments.

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