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Palynostratigraphy, Paleoenvironments and Kerogen Evaluation of the Campanian-Maastrichtian Enugu Shale, Anambra Basin, Nigeria

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Abstract

Palynological and palynofacies analyses were used to investigate the paleoenvironments and hydrocarbon potential of the Campanian-Maastrichtian Enugu Shale, Anambra Basin, Nigeria. The palynological analysis of twenty-three outcrop samples revealed diverse and abundant palynomorph assemblages, which consisted of pteridophyte spores 46.82%, angiosperms 36.9%, gymnosperms 1.48 %, algae 5.65%, dinoflagellate cysts 5.1%, fungal elements 1.17%, acritarchs 0.34%, microforaminiferal wall linings 0.01% and miscellaneous palynomorphs (Charred gramineae cuticle, incertae sedis) 2.53%. Seven informal palynological assemblage zones (PAZ) with corresponding eustatic sea level changes were delineated. Palynofacies analysis revealed four palynofacies types I to IV based on the percentage relative abundances of the sedimentary organic matter (SOM). Palynofacies types P-I and P-II suggest proximal fluvio-deltaic setting, while P-III and P-IV reflect a heterolithic-oxic shelf (proximal shelf) environment, indicative of deposition in an oxidizing condition. Results of the Spore Colour Index (SCI) and Thermal Alteration Index (TAI) analyses indicate that the Enugu shales are thermally immature.

Keywords: Enugu, Campanian, Maastrichtian, Palynomorphs, Phytoclasts

Introduction

Palynostratigraphy is the use of palynomorphs in sedimentary strata delineation, correlation and relative age dating. Several workers have used palynomorph distributions in stratigraphic differentiation of rock sequence over various geological periods (Umeji, 2010; Nwojiji *et al.*, 2013; El Atfy *et al.*, 2013; Onoduku & Okosun, 2014; Chiaghanam *et al.*, 2014; Stephenson, 2016). In addition, palynostratigraphic analysis can be used to establish stratigraphic boundaries. For example, the global Barremian/Aptian boundary of

the Maiolica Formation, Umbria-Marche Basin, Italy is defined by using the disappearance and appearance of the dinoflagellate cysts *Rhynchodiniopsis aptiana* and *Odontochitina operculata* respectively (Unida & Patruno, 2015). Moreover, local ranges of marine and non-marine palynomorphs allow correlation between facies and refining of zonation of total interval (Nichols & Jacobson, 1982; Sancay, 2005; Itam *et al.*, 2019).

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On the other hand, the palynofacies can be defined as the study of particulate organic matter assemblages (Boulter & Riddick, 1986) in terms of changes in the relative abundance of various types (e.g., palynomorphs, bioclasts, phytoclasts, amorphous organic matter (AOM). Palynofacies analysis has been used to understand the depositional regime, thermal maturation and paleoenvironment of many sedimentary basins in the world. For example, the Vale of Glamorgan Basin, South Wales (Davies *et al.*, 1991), the Mesopotamian Basin, southern Iraq (Al-Ameri *et al.*, 1999), the Keta Basin, Ghana (Apaalse & Atta-Peters, 2013), the Northern Carnarvon Basin, NW Australia (Dixon, 2013), the Larsen Basin, James Ross Island, Antarctica (Carvalho *et al.*, 2013), the Ghadames Basin, north-west Libya (El Diasty *et al.*, 2017).

In addition, palynofacies data helped to detect small-scale cyclic palaeoenvironmental variations that allow division of palaeoenvironmental cycle into a number of distinct units, which are not evident in the sedimentological data (Waterhouse, 1995). Besides, some important palynofacies parameters are employed as indicators of proximal–distal trends, overall water depth and sediment accumulation rate (Tahoun *et al.*, 2013; Thomas *et al.*, 2015). Moreover, climate transition can be studied at high resolution with the help of palynofacies analysis. For instance, the Early Permian movement of Gondwana away from the South Pole resulted in the shift from a post-glacial Carboniferous flora to a temperate Permian flora, which was detected by palynofacies analysis (Wheeler & Götz, 2016).

Spore coloration and visual kerogen analysis are used to assess the thermal maturation and petroleum potential of source rocks (Zobaa *et al.*, 2013; Makled and Baioumi 2013; Thakur and Dogra 2011; Ribeiro *et al.*, 2013; El Atfy 2018).

A good number of researchers have studied the lithofacies, age and paleoenvironments of the Campanian-Maastrichtian sedimentary fill of Anambra Basin. These include Obaje *et al.* (1999), Umeji (2006), Ogala *et al.* (2009), Onyekuru & Iwuagwu (2010), Onuigbo *et al.* (2012a), Onuigbo *et al.* (2012b), Soronnadi-Ononiwu *et al.* (2012),

Chiaghanam *et al.* (2013a; 2013b), Adeigbe & Salufu (2009) and Ehinola (2010).

However, few works have been done on the palynofacies of the Anambra Basin (Obok-Ikuenobe *et al.*, 2005; Chiaghanam *et al.*, 2014; Maju-Oyovwikowhe & Malomi 2019; Onyedikachi, 2019; Lucas, 2018; Oke & Onoduku, 2018; Durugbo & Ogundipe, 2019). Dinoflagellate cysts study have received very little attention in the study area even though they have proven to be very useful in the interpretation of depositional environments (Li and Habib 1996). The present study, therefore, attempts to combine the non-marine and marine polymorphs of the outcrop samples from the Enugu Shale, Anambra Basin, for age dating, paleoenvironmental reconstruction and evaluation of source rocks potentials.

Geological setting and stratigraphy

The Enugu Shale belongs to the Nkporo Group of the Anambra Basin (Figs. 1 and 2). The Nkporo Group is considered the oldest sedimentary unit in the basin (Nwajide, 1990). Deposited in Late Campanian, it is composed of the Nkporo Shale, Owelli Sandstone and Enugu Shale (Reyment, 1965; Obi *et al.*, 2001). The Nkporo Group is overlain by the Mamu Formation which was deposited in the Early Maastrichtian (Kogbe, 1989; Obi, 2000). It comprises of succession of siltstone, shale, coal seams and sandstones (Kogbe, 1989). Furthermore, the Mamu Formation is overlain by the Ajali sandstone (Maastrichtian) which is mainly unconsolidated coarse to fine grained, loose, very fine-grained sand, mudstone and siltstone (Reyment 1965; Kogbe 1989; Nwajide 1990). The diachronous Nsukka Formation (Maastrichtian-Danian), which is also known as the Upper Coal Measure (Reyment, 1965; Salami, 1990; Obi, 2000; Durugbo, 2016), overlies the Ajali Sandstone. In addition, overlying the Nsukka Formation is the Imo Shale, which is of Paleocene age (Nwajide, 1990; Durugbo, 2013).

The Nsukka Shale comprises clayey shale with occasional ironstone and thin sandstone beds in which carbonized plants remains may occur (Kogbe, 1989). The youngest sediments are the

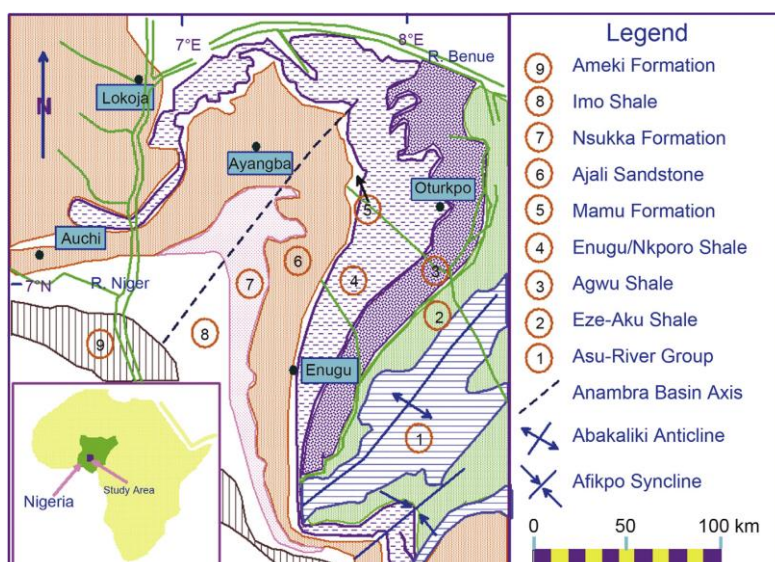


Figure 1. Geological map of the Anambra Basin and location of study site (modified after Nton & Bankole, 2013)

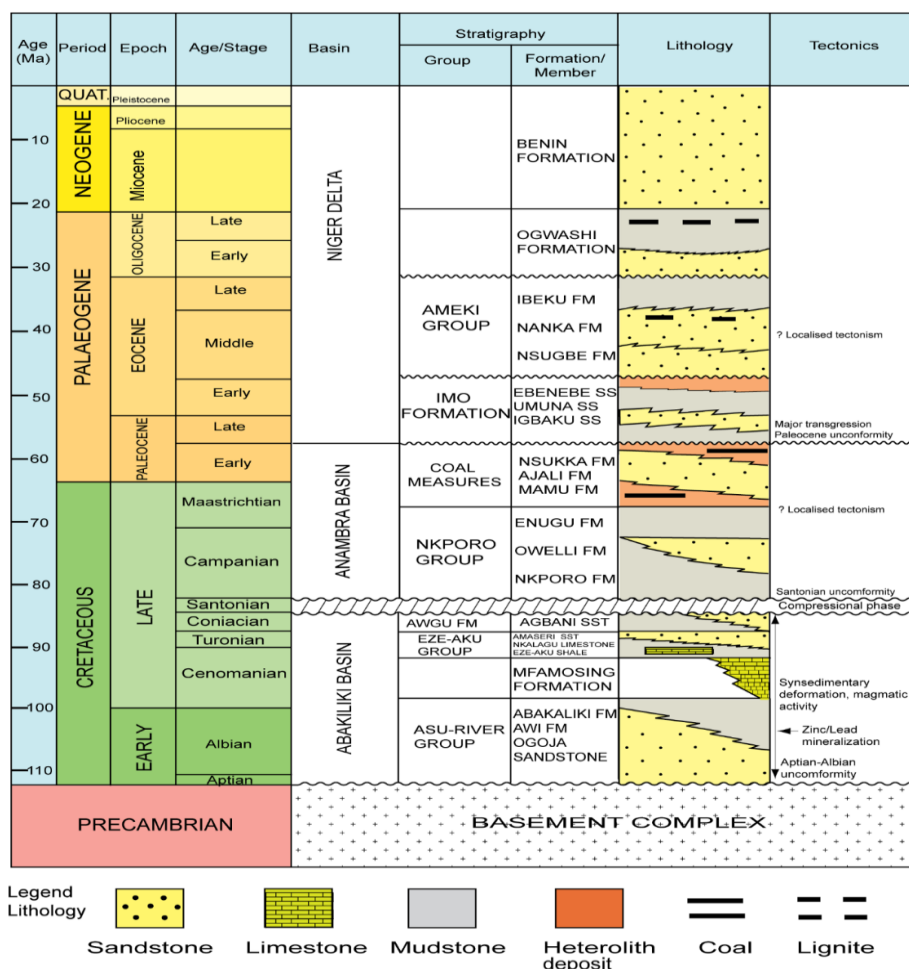


Figure 2. Lithostratigraphic framework for the Early Cretaceous-Tertiary period in southeastern Nigeria (after Nwajide, 1990).

Eocene Ameki Group, deposited during a regressive phase (Obi, 2000).

The Enugu Formation consists of grey, blue or dark shale, occasional white sandstones and striped sandy shale beds (Nwajide, 2013; Fig. 2). The Formation has its type locality at the Enugu Municipality, with an area coverage that stretches north to Ikem-Ihandiagu area, and southwards to Awgu area (Nwajide, 2013). The formation consists mainly of shales, with two distinguishable sandstone bodies, the Otobi and the Okpaya Sandstones, which are regarded as members of the formation (Nwajide, 2013). The formation is restricted to the central and northern parts of the Anambra Basin, with a thickness of about 300ft. The Enugu shale was assigned Campanian to Lower Maastrichtian, based on the diagnostic species of palynomorphs such as *Cingulatisporites ornatus* and *Tricolpites tienebaensis* (Reyment, 1965; Whiteman, 1982; Soronnadi-Ononiwu *et al.* 2012).

Materials and methods

Twenty-three outcrop samples were collected at 1m intervals from the Enugu Shale exposed around Trans Ekulu Golf area along the Enugu-Onitsha expressway (Fig. 3). The coordinates of the location N06 28.224 and E007 28.233 were taken with Garlux S70 GPS equipment. The surface was scrapped to avoid weathered and contamination with recent materials. The samples were collected with sterile nylon and deposited at the Biological Sciences laboratory of Redeemer's University Ede, Osun State prior to sample preparation. The samples were subjected to standard palynological preparation procedures (Faegri & Iversen, 1989) involving disaggregation and removal of carbonates and silicates with hydrochloric and hydrofluoric acids respectively. The samples were further treated with hot Hydrochloric acid (HCL) and wet-sieved over a 5-micro mesh polypropylene sieve. Zinc Bromide SG 2.2 was used for effective removal of silt and clay particles. Each residue was oxidized using concentrated nitric acid (HNO₃) and prepared for study as strewn mounts using Loctite. The slides for palynofacies were not oxidized or

sieved because the organic components were identified by colors (Obboh-Ikuenobe *et al.*, 1998). The slides were analyzed and all the palynomorphs present (pollen, spores, fungal remains, algae, dinoflagellate cysts and foraminiferal linings) were recorded and the totals and percentages of the different groups calculated (Table 1). The distribution of the different palynomorph groups were plotted on Tilia pollen diagram (Fig. 4). Photomicrographs of index species (Plates I -VI) were taken with a Zeiss Axioskop 2 microscope with an attached AxioCam 1Cc 1 Camera at the Palynology laboratory of the Evolutionary Studies Institute, University of the Witwatersrand, Johannesburg, South Africa. The slides, residues, unprocessed samples, and duplicate prints are housed in the palynological collections of the Biological Sciences Department, Redeemer's University, Ede, Osun State, Nigeria. The different dispersed organic matter and palynomorph groups identified are pollen and spores, fungal remains, freshwater algae, marine palynomorphs, structured phytoclasts (wood, cuticles, parenchyma), unstructured phytoclasts (resins, comminuted and degraded fragments, black debris, and amorphous organic matter (Batten, 1982; Obboh-Ikuenobe *et al.*, 2005; Durugbo, 2016). Visual colour analysis was carried out using Deltoidospora/Cyathidites as the standards (Ibrahim *et al.*, 1997; Makled *et al.*, 2013; Atta Peters & Achaegakwo, 2016) based on comparison with the Munsell colour standards (Pearson 1984 in Pross *et al.*, 2007).



Figure 3. The sampled sites around Trans Ekulu Golf area along the Enugu-Onitsha express way

Results

A total count of six thousand, eight hundred and sixty-three (6,863) palynomorphs were recorded (Table 1). These composed of spores (46.42 %), *Palmae* (22.2%), other pollen (14.7%), gymnosperms (1.28 %), dinoflagellate cysts (5.1%), microforaminiferal wall linings (0.01%), acritarchs (0.34%), algae (5.45%), fungal elements (1.17%) and miscellaneous palynomorphs (Charred gramineae cuticle, *incertae sedis*) (3.33%). The

depth-by-depth occurrences of these palynomorphs according to their ecological groups are displayed on Tilia™ Graph (Fig. 4).

Palynological assemblage zones

As no palynological zonation scheme exists for the Nigerian Cretaceous, the seven informal microfloral assemblage zones resulting from CONISS (Fig. 4) are hereby discussed

Table 1: Total Abundances of palynomorphs recovered from Enugu Shale samples

Sample Number	Total <i>Palmae</i>	Total Gymnosperms	Total Other pollen	Total spores	Total dinoflagellate cysts	Microforaminiferal wall-linings	Acritarch	Total Algae	Miscellaneous palynomorphs	Fungal elements
Sample 1	18	2	10	53	5	0	2	6	4	2
Sample 2	44	1	19	45	4	1	2	3	29	14
Sample 3	66	2	39	89	6	0	0	8	28	14
Sample 4	73	0	59	91	6	0	1	17	17	8
Sample 5	39	10	19	103	7	0	2	1	8	4
Sample 6	92	5	46	200	24	0	4	23	18	9
Sample 7	56	4	40	127	16	0	1	3	14	7
Sample 8	37	4	44	119	14	0	0	2	1	0
Sample 9	130	8	69	278	26	0	4	15	1	0
Sample 10	136	9	80	263	27	0	0	24	2	0
Sample 11	18	2	5	39	3	0	1	5	0	0
Sample 12	54	4	21	90	5	0	0	39	0	0
Sample 13	46	2	21	100	28	0	2	36	20	10
Sample 14	13	3	9	33	7	0	0	4	3	1
Sample 15	72	3	48	145	52	0	1	14	6	2
Sample 16	88	2	67	192	11	0	0	24	16	7
Sample 17	73	1	77	211	14	0	1	27	0	0
Sample 18	82	2	76	218	34	0	0	11	4	2
Sample 19	112	9	89	192	17	0	1	28	3	0
Sample 20	76	1	20	119	2	0	0	14	0	0
Sample 21	28	4	27	72	0	0	0	9	0	0
Sample 22	81	6	48	137	25	0	0	33	0	0
Sample 23	143	4	76	270	16	0	1	28	2	0
Total	1577	88	1009	3186	349	1	23	374	176	80
Percentage	22.97	1.28	14.70	46.42	5.1	0.01	0.34	5.45	2.56	1.17

A marginal marine environment with frequent freshwater incursion is suggested for this interval due to the association of dinocysts of dominantly gonyaulacean affinity co-occurring with common palm pollen and freshwater algae.

Palynological assemblage zone PAZ B (20.0-18.0 m)

The microfloral signature within this short interval is dominated by palm pollen in association with *Constructipollenites ineffectus*, *Buttinea andreevi*, *Echitriporites simpliformis*, *Proteacidites dehaani*, *Ericipites* sp., *Ephedripites multicostatus*, *Auriculiidites reticulatus*, *Ephedripites multicostatus* and *Inaperturites cristatus*. Others are *Monocolpopollenites sphaeroidites*, *Syncolporites marginatus*, *Syndemicolpites typicus*, *Sapotaceoidaepollenites* sp. The pteridophyte spores in this zone are *Cyathidites minor*, *C. australis*, *Deltoidospora* sp., *Laevigatosporites ovatus*, *Zlivisporites blanensis*, *Rugulatisporites caperatus*, *Cingulatisporites ornatus*, *Distaverrusporites simplex*, *Stereisporites* sp., *Ariandnesporites spinosus*, *A. nigeriensis*, *Verrucatosporites* sp. and *Rugulatisporites caperatus*. The dinoflagellate suite within this zone was low *Coronifera oceanica*, *Dinogymnium nelsoense*, *Selenopemphix* sp., *Spiniferites ramosus*, *Cleistosphaeridium* sp., *Diphyes spinulum*, *Exochosphaeridium bifidum*, *Florentinia ferox*, *Florentinia* sp., *Subtilisphaera* sp., *Spiniferites* sp., and the acritarch *Leiosphaeridia* sp., in association with common freshwater algae *Botryococcus braunii*, spot records of *Concentricytes* sp. and *Pediastrum* sp. There are rare fungal elements and Charred Graminae Cuticle. A marginal marine environment with frequent freshwater incursion is suggested for this interval due to the association of dinocysts of dominantly gonyaulacean affinity co-occurring with common palm pollen and freshwater algae.

Palynological assemblage zone PAZ C (18.0-14.0 m)

Pteridophyte spores especially *Cyathidites minor*, *C. australis*, *Laevigatosporites ovatus*,

Deltoidospora spp., *Cingulatisporites ornatus*, *Rugulatisporites caperatus*, *Zlivisporites blanensis* and *Ariandnesporites nigeriensis* dominated the assemblage within this section. These occurred with moderate records of *Constructipollenites ineffectus*, *Longapertites marginatus*, *L. microfoveolatus*, *L. vanendeenburgi*, *Echitriporites trianguliformis*, *Ephedripites multicostatus*, *Ericipites* sp., *Buttinea andreevi*, *Retidiporites magdalenensis*, *Auriculopollenites reticulatus*, *Proteacidites dehaani*, *Psilamonocolpites* sp., *Sapotaceoidaepollenites* spp., *Syncolporites marginatus*, *Monocolpites marginatus*, and *Milfordia jardenei*.

The dinocysts were represented by common *Coronifera oceanica*, *Cleistosphaeridium* sp., *Florentinia* sp., *Diphyes spinulum* and *Operculodinium* sp., in association with low records of freshwater algae *Botryococcus braunii*, *Pediastrum* sp. and *Azolla cretacea*.

A marginal marine environment with frequent freshwater incursion is suggested for this interval due to the association of dinocysts of dominantly gonyaulacean affinity co-occurring with common palm pollen, freshwater algae and low records of fungal elements.

Palynological assemblage zone PAZ D (14.0-11.0 m)

There was a noticeable reduction in the recovered palynomorphs within this section *Longapertites* spp., *Buttinea andreevi*, *Monocolpopollenites sphaeroidites*, *Ephedripites multicostatus*, *Constructipollenites ineffectus*, *Zlivisporites blanensis*, *Rugulatisporites caperatus*, *Cingulatisporites ornatus*. Others are *Distaverrusporites simplex*, *Cyathidites minor*, *C. australis*, *Deltoidospora* spp., *Stereisporites* sp., *Monocolpites marginatus*, *Milfordia jardenei*, *Retidiporites magdalenensis*, *Echitriporites simpliformis* and *E. longispinosus*. Moreover, *Auriculiidites* spp., *Ariandnesporites spinosus*, *A. nigeriensis*, *Foveotriletes margaritae* and *Tubistephanocolporites cylindricus* occurred with

records of the dinoflagellate cysts *Florentinia* sp., *Oligosphaeridium* complex, *Coronifera oceanica*, *Dinogymnium acuminatum*, *D. undulosum*, *Diphyes* sp., *Paleocystodinium gabonensis*, *Andalusiella* sp. and *Cleistosphaeridium* sp. There are abundant records of freshwater algae *Azolla cretacea* together with sparse *Botryococcus braunii*, *Pediastrum* sp., and *Concentricytes* sp.

Palynological assemblage zone PAZ E (11.0-7.0 m)

The assemblage within this section is dominated by *Constructipollenites ineffectus*, *Echitriporites trianguliformis*, *Longapertites marginatus*, *L. microfoveolatus*, *L. chlonovae*, *L. vanendeenburgi*, *L. reticulatus*, *Ericipites* sp., *E. longispinosus* and *Ephedripites multicostatus*. Other dominant palynomorphs are *Monocolpites marginatus*, *Gnetaceaepollenites* sp., *Proteacidites dehaani*, *P. sigalii*, *Psilamonocolpites medius*, *Buttinea andreevi*, *Auriculopollenites reticulatus*, *Aquilapollenites* sp., *Monocolpopollenites sphaeroidites* and *Syncolporites marginatus*. Moreover, *Spinizonocolpites baculatus*, *Tubistephanocolporites cylindricus*, *Milfordia jardenei*, *Sapotaceoidaepollenites* spp., *Spinizonocolpites kotschiensis* and *Liliacidites nigeriensis* occurred abundantly. Spot occurrences of *Syndemicolpites typicus*, *Syncolporites lisame*, *Grimsdalea magnaclavata*, *Mauritidites lehmanii*, in association with pteridophyte spores *Cyathidites minor*, *C. australis*, *Zlvisporites blanensis*, *Rugulatisporites caperatus*, *Cingulatisporites ornatus* *Distaverrusporites simplex*, *Deltoidospora* spp., *Stereisporites* sp. and *Ariandnesporites nigeriensis*. Others are *Foveotrilletes margaritae*, *Laevigatosporites ovatus*, *Concavissimisporites* sp., *Cicatricosisporites* sp. and *Leiotrilletes* sp. These land derived palynomorphs occurred together with records of the dinoflagellate cysts *Coronifera oceanica*, *Cleistosphaeridium* sp., *Exochosphaeridium bifidum*, *Canningia* sp., *Florentinia* sp., *Diphyes spinulum*, *Thalassiphiora* sp., *Chytroespaeridia* sp., *Spiniferites ramosus*, *Spiniferites* sp., *Hystriochosphaeridium* sp., spot records of *Florentinia ferox*, *Dinogymnium acuminatum*, *Diphyes spinulum*, *Cribroperidinium*

sp., *Glaphyrocysta* sp., *Lingulodinium* sp., *Polysphaeridium* sp. Also recovered within this section were low records of freshwater algae *Botryococcus braunii*, *Pediastrum* sp., and *Azolla cretacea* and fungal elements.

Palynological assemblage zone PAZ F (7.0-3.0 m)

The pteridophyte spores *Cyathidites minor*, *C. australis* and *Deltoidospora* spp. dominated the assemblage within this section. These occurred with moderate records of *Constructipollenites ineffectus*, *Longapertites marginatus*, *L. microfoveolatus*, *L. vanendeenburgi*, *Echitriporites trianguliformis*, *Ephedripites multicostatus*, *Proteacidites dehaani*, *Syncolporites marginatus*, *Buttinea andreevi*, *Auriculopollenites reticulatus*, *Sapotaceoidaepollenites* spp., *Ericipites* sp., and *Psilamonocolpites* sp.

Rare dinoflagellate cysts, low records of freshwater algae *Botryococcus braunii*, *Pediastrum* sp. and *Azolla cretacea*. Moreover, there is spot occurrences of *Ariandnesporites nigeriensis*, *Zlvisporites blanensis*, *Rugulatisporites caperatus*, *Cingulatisporites ornatus*, *Distaverrusporites simplex*, *Stereisporites* sp., *Ariandnesporites nigeriensis*, *Foveotrilletes margaritae*, *Laevigatosporites ovatus*, *Concavissimisporites* sp., *Cicatricosisporites* sp., and *Leiotrilletes* sp.

Palynological assemblage Zone PAZ G (3.0-1.0 m)

This is the youngest interval of the studied Enugu Shales. There was a marked reduction in palynoflora within this assemblage zone compared to the underlying assemblage zones. The microfloral assemblage is characterized by common *Constructipollenites ineffectus*, *Longapertites marginatus*, *L. microfoveolatus*, *L. vanendeenburgi*, *Longapertites* spp., *Retidiporites magdalenensis*, *Echitriporites trianguliformis*, *E. longispinosus*, *Proteacidites dehaani* and *P. sigalii*. Other common palynomorphs are *Buttinea andreevi*, *Auriculopollenites reticulatus*, *Aquilapollenites* sp., *Ephedripites multicostatus*, *Monocolpites marginatus*, *Ericipites* sp.,

Monocolpopollenites sphaeroidites, *Syncolporites marginatus*, *Spinizonocolpites baculatus*, *Milfordia jardenei*, *Periretisyncolpites magnosagenatus*, *Sapotaceoidaepollenites* spp., *Foveomonocolpites bauchiensis*. The pteridophyte spores comprises of *Cyathidites minor*, *C. australis*, *Zlivisporites blanensis*, *Rugulatisporites caperatus*, *Cingulatisporites ornatus*, *Distaverrusporites simplex*, *Deltoidospora* spp. and *Stereisporites* sp. Moreover, *Ariandnesporites nigeriensis*, *Foveotriletes margaritae*, *Laevigatosporites ovatus*, *Concavissimisporites* sp., *Cicatricosisporites* sp., *Leiotriletes* sp., which occurred with records of the dinoflagellate cysts *Coronifera oceanica*, *Cleistosphaeridium* sp., *Exochosphaeridium bifidum*, *Diphyes spinulum*, *Florentinia* sp., *Chytroesphaeridia* sp., *Spiniferites ramosus*, *Spiniferites* sp.. In addition, there are spot records of *Dinogymnium euclaense*, *Andalusiella* sp., *Lingulodinium* sp., and *Thalassiphora* sp., and common records of freshwater algae *Botryococcus braunii*, *Pediastrum* sp., and *Azolla cretacea*.

Paleoenvironments and kerogen evaluation

Palynofacies analysis

Combaz (1964) was first author to use the term palynofacies to describe acid-resistant organic matter in sediments. Thereafter, other authors have used the concept in palaeoenvironmental and depositional interpretation (Hughes and Moody-Stuart, 1967; Batten, 1973, 1981, 1982; Boulter & Riddick, 1986; Powell *et al.*, 1990). The palynofacies classification terms used herein is after the work of Tyson (1995) which are amorphous organic matter (AOM), phytoclasts and palynomorphs. Palynomorphs include all spores, pollen, freshwater algae and fungal remains (regarded as terrestrial palynomorphs), dinocysts and microforaminiferal lining (regarded as marine palynomorphs). Phytoclasts include structured and degraded/unstructured terrestrial plant fragments (cuticles, wood tracheid and cortex tissue), and opaque (black debris). AOM includes all particulate organic components that appear structureless at the scale of light microscopy, including bacterially derived AOM, resinous and amorphous products of the diagenesis of macrophyte tissues (Atta-Peters *et al.*, 2015). The

relative abundance of the palynofacies element recovered from the samples are recorded (Table 2).

Palynofacies Types

Cluster analysis of the percentage composition of palynofacies elements revealed three clusters: A, B and C (Table 2, Fig. 6). Cluster B is further subdivided into two sub-clusters B1 and B2 (Fig 6).

Palynofacies Type I (P-I)

This palynofacies type, which corresponds to cluster A is contained in the samples at depths 23 m, 19 m, 17 m, 10 m, 6 m and 3 m respectively (Figs. 5 and 6). It is dominated by structured phytoclasts (81%). This is followed by degraded/unstructured phytoclasts (7%), terrestrial palynomorphs (5%), opaque phytoclasts (3%), fungal remains (2%), freshwater algae (1%) and AOM (1%) with the absence of marine palynomorphs (Fig. 6).

Palynofacies Type II (P-II)

Palynofacies type II is equivalent to B1, encountered at sample depths 21 m, 20 m, 12 m, 8 m, 7 m, 2 m and 1 m (Figs. 5 and 6). The palynofacies type II is typified by relative abundant of structured phytoclasts about 64% and followed by degraded/unstructured phytoclasts (24%), freshwater algae (6%), opaque phytoclasts (4%), AOM (1%) and terrestrial palynomorphs (1%). Marine palynomorphs and fungal remains are absent (Fig. 6).

Palynofacies Type III (P-III)

Palynofacies type III is equivalent to cluster B2 and represented in samples at depths 22 m, 16 m, 14 m, 13 m, 11 m, 5 m and 4 m (Figs. 5 and 6). Palynofacies type III is distinguished from other types by the dominance of structured phytoclasts about 63% and followed by structureless phytoclasts (16%), opaque phytoclasts (7%), terrestrial palynomorphs (6%), freshwater algae (5%) and 1% of AOM, fungal remain and marine palynomorph (Fig. 6).

Palynofacies Type IV (P-IV)

Palynofacies type IV corresponds to cluster C and represented in samples at depths 18 m, 16 m and 9 m (Figs. 5 and 6). The palynofacies type is

Table 2: Relative Abundance of palynofacies element recovered from Enugu Shale samples

Sample number	Palynomorphs	Marine Palynomorphs	Freshwater algae	Fungal remains	Structured phytoclasts	Degraded/unstructured phytoclasts	Opaque	Amorphous organic matter	Total
Sample 1	10	3	6	3	184	82	10	2	300
Sample 2	7	1	4	4	198	72	13	1	300
Sample 3	15	0	3	6	242	20	10	4	300
Sample 4	8	1	8	3	213	48	15	4	300
Sample 5	18	2	2	3	211	50	12	2	300
Sample 6	17	7	15	4	205	32	16	4	300
Sample 7	10	5	1	1	178	82	20	3	300
Sample 8	5	3	6	0	197	72	15	2	300
Sample 9	22	8	12	2	168	74	10	4	300
Sample 10	33	7	16	1	207	24	10	2	300
Sample 11	7	1	6	1	211	58	15	1	300
Sample 12	2	0	17	1	193	73	12	2	300
Sample 13	17	6	20	4	185	56	10	2	300
Sample 14	10	1	6	0	200	74	8	1	300
Sample 15	22	13	5	1	176	66	12	5	300
Sample 16	18	3	15	3	188	49	22	2	300
Sample 17	13	2	14	0	215	24	30	2	300
Sample 18	27	6	6	1	156	78	23	3	300
Sample 19	32	5	17	0	220	17	7	2	300
Sample 20	4	0	7	0	196	70	23	0	300
Sample 21	5	0	3	1	184	85	21	1	300
Sample 22	10	6	18	2	188	61	13	2	300
Sample 23	33	3	15	4	196	32	15	2	300

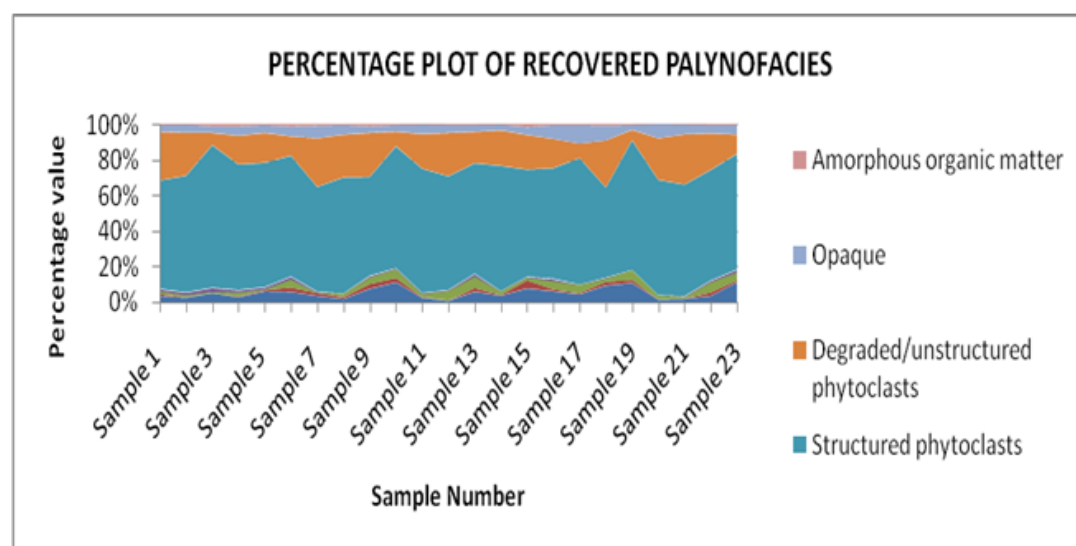


Figure 5. Percentage relative abundances of palynomorphs, phytoclasts, opaques and AOM

characterized by high amount of structured phytoclasts of about 51%, which is followed by structureless phytoclasts (27%), terrestrial palynomorphs (9%), opaque phytoclasts (8%), 2%

of freshwater algae and marine palynomorphs and 1% of AOM. Fungal remains is absent in this palynofacies type (Fig. 6).

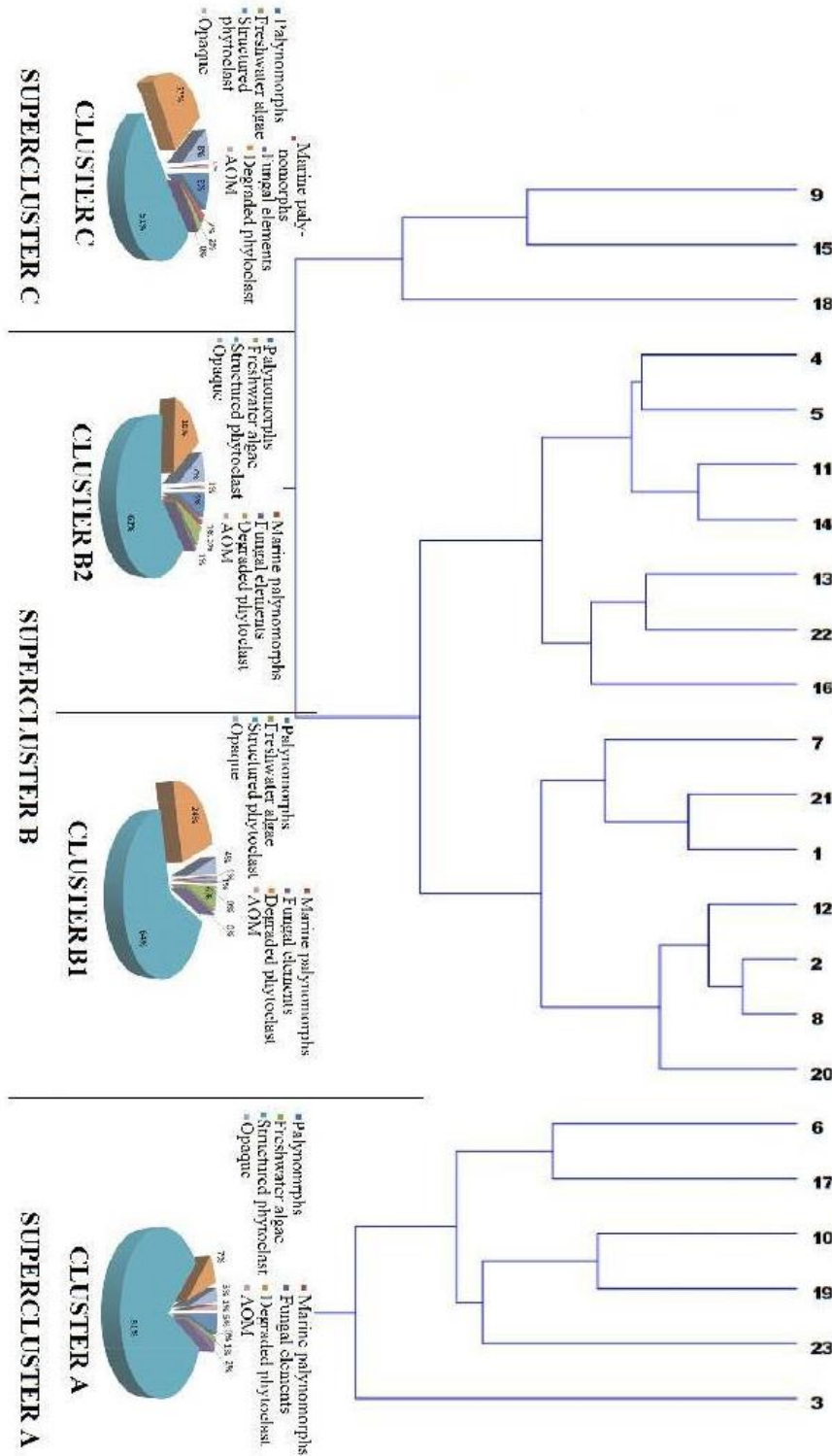


Figure 6. Cluster Analysis shows grouping samples into palynofacies types

Hydrocarbon potentials

Spore color index (SCI) and thermal alteration index (TAI)

Miospore color determinations in all Enugu outcrop samples are restricted to smooth, thin-walled species such as those of *Deltoidospora adriennis* and *Cyathidites minor*. Thermal maturation estimations (Pearson 1984 in Pross *et al.*, 2007)

were made, the colour chart correlated with corresponding thermal alteration index (TAI) values (e.g., Traverse, 2007; Batten, 1980). Spore and pollen exine colors in the studied Enugu outcrop samples collectively ranged from colorless to golden yellow correlated to ≈ 1 to 1^+ TAI values (Fig. 7), indicating negligible chemical change in the path of maturity (e.g., Batten, 1980).

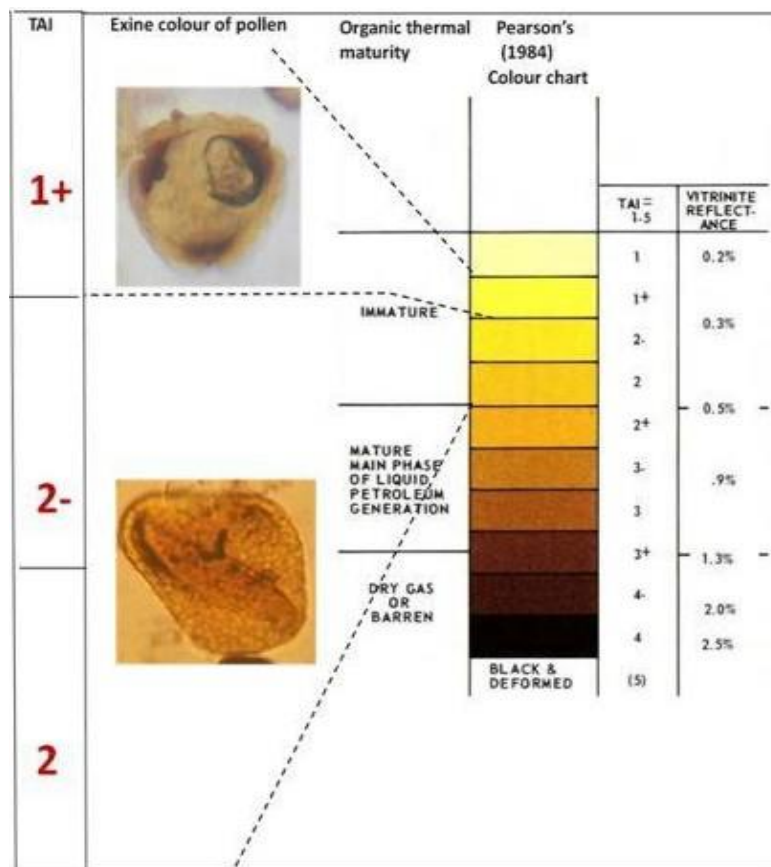


Figure 7. Sporomorph colour index for the Enugu Shale (after Pearson 2007)

Discussion

The Campanian/Maastrichtian boundary

Many reports have highlighted the discrepancies in delineating the Campanian/Maastrichtian boundary. For example, Muller *et al.* (1987) had associated the top occurrence of *Droseridites senonicus* and *Buttinea andreevi* / *Proteacidites dehaani* with the base and top of their Superzone VI (*Auriculidites reticularis*, *Crassitricolporites subprolatus* and *Proteacidites dehaani* zones) of South America respectively, which is dated Campanian to Maastrichtian. In addition, Eisawi & Shrank (2008) working in southern Sudan could not

confidently define the Campanian /Maastrichtian boundary. They had utilized the first downhole occurrences of *Auriculidites reticulatus*, *Gabonispuris vigourouxii*, *Mauritidites lehmanii*, *Monocolpopollenites sphaeroidites* and *Spinizonocolpites kotschiensis* to define the top of their Assemblage Zone II, which they dated Campanian to Maastrichtian. Furthermore, the FDO: *Ariadnaesporites spinosus*, *Proteacidites sigalii* and *Retidiporites magdalenensis* within the Maastrichtian are also reported. However, in the present study, FDO: *Spinizonocolpites kotschiensis* that occurred at sample depth 9 m marked base of

Maastrichtian. This species is also encountered in samples at depths 16 and 17 meters. In addition, FDO: *Monocolpopollenites sphaeroidites* encountered at Sample depth 5 m and also occurred in sample depths 9 m, 11 m, 12 m, 13 m, 15 m, 16 m, 17 m, 18 m 22 m and 23 m.

Palynology and paleoenvironmental deductions

The preponderance of palm pollen *Longapertites* spp., *Mauritidites* spp., *Proxapertites* spp., and *Retidiporites magdalenensis* proves that the studied area falls within the Late Cretaceous Palmae province of Herngreen (1980), Herngreen & Chlonova (1981). Furthermore, the paleogeographic position of the studied area close to the paleo-equator further conformed this, as reported by Eisawi & Shrank (2008) for the Melut Basin, southeast Sudan. Other workers had reported this common recovery of palmae pollen in southeastern Nigeria (Salami, 1990; Umeji and Nwajide, 2007; Umeji & Edet, 2008; Durugbo, 2013, 2016). According to Eisawi and Shrank (2008), the common records of structured phytoclasts with low amorphous organic matter indicates deposition in high energy fluvial settings close to the vegetation sources. Again, the presence of the spores of water ferns (*Ariadnaesporites nigeriensis*, *A. spinosus* and *Gabonisporis vigourouxii* together with the Hepaticae (*Zlivisporis blanensis*) indicates the development of tropical swampy ecosystems within a predominantly fluvial setting during the period under study. Ukaegbu & Akpabio (2009) had associated high concentration of terrestrially derived organic matter as recorded in the Enugu shale to indicate proximity to the paleoshoreline. On the other hand, high concentration of marine derived AOM is indicative of a distal location (Obboh-Ikuenobe *et al.*, 1998). Notably, ephedroid pollen occurred sparsely as they have been associated with dry environments (Salard & Dejax, 1991; Eisawi & Shrank, 2008).

The common presence of gonyauloid dinoflagellate cysts in the present studied site indicates a pronounced marine transgression than the Okaba coal mine site earlier studied by Umeji (2005) in which she recorded few dinoflagellate cysts. Adeigbe & Salufu (2009) had attributed the presence of extraformational clasts within the Enugu Shale to

pronounced fluvial incursion which could have accounted for the preponderance of *Azolla cretacea* in the present samples (Tucker, 1996; Adeigbe & Salufu, 2009), this is confirmed by the common records of freshwater algae. The fissility and the fine nature (Grain size) of the Enugu Shale as indicated by the field data suggest that Enugu Shale was deposited below the wave base, accumulated in relatively low energy environment i.e. in a distal to proximal lagoon (Amaral & Pryor, 1974; Adeigbe & Salufu, 2009). The light grey colour of the shale shows that Enugu Shale was deposited on the surface of the basin where oxidation could take place (Dapple, 1974). The thick, dark grey to black shales interbedded with siltstones (Fig. 3) are interpreted as shallow to open marine deposits. The presence of marine dinocysts and burrows of the Cruziana-Zoophycus ichnofacies support this (Pemberton *et al.*, 2001). The succession exhibits “sandier (coarsening) upward” characteristics often interpreted as “shoaling upward” or progradation (Reading & Collinson, 1996). The heterolithic units (sandstone/siltstone/shale) that overlies the thick shale units of the Enugu Formation can be interpreted as the sedimentary deposits of the lower delta front (distal bar) to near-shore environment with a considerable marine influence. The heterolithic units are thus assigned to marginal marine (near-shore to shoreface) environment. The rippled and parallel laminated sandstones of the Mamu Formation are interpreted as tidal/fluvial/distributary channel deposits in marsh and coastal swamp setting.

Tschudy (1973) had recovered *Cicatricosisporites stoveri*, *Stereisporites* spp., and several *Aquilapollenites* sp., from the Upper Campanian Judith River Formation in North Central Montana. Salami (1990) had recovered majority of the palynomorphs from the “Lower Coal Measures” deposits from the Anambra Trough southeastern Nigeria, which he dated Campanian-Maastrichtian. They include *Distaverrusporites simplex*, *Cingulatisporites ornatus*, *Rugulatisporites caperatus*, *Ariadnaesporites spinosus*, *Zlivisporites blanensis*, *Buttinea andreevi*, *Monocolpopollenites sphaeroidites*, *Monocolpites marginatus*, *Milfordia jardenei*, *Aquilapollenites minor*, *Retidiporites*

magdalenensis, *Gnetaceapollenites* sp., *Ephedripites* sp., *Echitriporites simpliformis*, *E. longispinosus*, *Auriculiidites* spp., *Ariandnesporites spinosus*, *A. nigeriensis*, *Foveotriletes margaritae*, *Tubistephanocolporites cylindricus*, with records of the dinoflagellate cysts *Florentinia* sp., *Oligosphaeridium complex*, *Coronifera oceanica*, *Dinogymnium acuminatum*, *D. undulosum*, *Diphyes* sp., *Paleocystodinium gabonensis*, *Andalusiella* sp., *Cleistosphaeridium* sp., and common records of freshwater algae *Botryococcus* spp., *Pediastrum* spp., and *Azolla cretacea*.

A Late Campanian – earliest Maastrichtian age of the basal part of the Istebna Formation (Zarovjanka section, Table 3) is confirmed by the presence of *Cerodinium diebelii* and *Palaeocystodinium golzowense*. The first occurrence of *C. diebelii* and its related forms (Ypes 2001) is close to the Campanian/ Maastrichtian boundary (Antonescu *et al.* 2001; Kirsch 1991; Roncaglia 2002).

Palynofacies, Palaeoenvironmental deductions and hydrocarbon potential

Palynological and palynofacies data retrieved from Enugu outcrops indicate that the samples plot within the fields I and III on the AOM - phytoclasts

- palynomorphs (APP) ternary diagram (Fig. 8), representing highly proximal shelf or basin and heterolithic-oxic shelf (proximal shelf) with abundant phytoclasts respectively (Tyson, 1993).

Palynofacies type I (P-I) and type II (P-II) plot in field I of Tyson's APP ternary diagram, which indicates deposition in a highly proximal shelf or basin condition. It is distinguished by very good structured phytoclasts preservation and low occurrence of degraded phytoclasts. There are very little occurrences of terrestrial palynomorphs, opaque phytoclasts, fungal remain, freshwater algae and AOM and no occurrence of marine palynomorphs in this palynofacies type. On the other hand, palynofacies type III (P-III) and type IV (P-IV) plot in field III of Tyson's ternary diagram that indicates heterolithic-oxic shelf (proximal shelf) condition. This is characterized by moderate to good structured phytoclasts preservation in association with low to moderate occurrence of degraded phytoclasts. There are little to fair occurrence of opaque phytoclasts, terrestrial and marine palynomorphs, freshwater algae, AOM and fungal remain with absence of fungal remain in palynofacies type IV (P-IV). Presence of marine palynomorphs in P-III and P-IV indicates marine incursion into the land at this period.

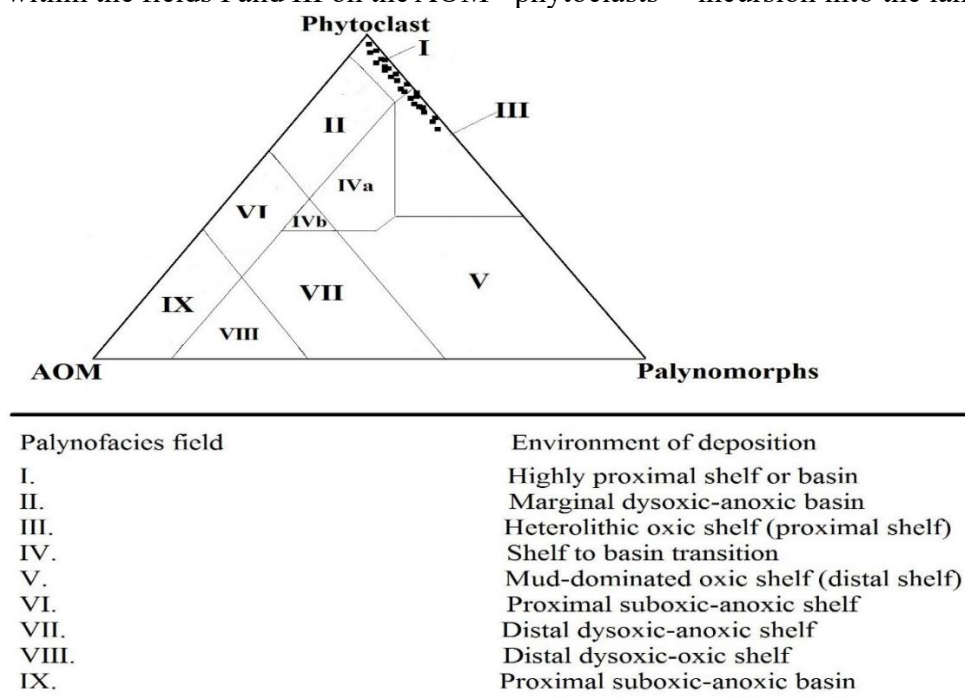


Figure 8. APP ternary palynofacies diagram used for palaeoenvironmental interpretation (Modified after Tyson, 1993)

The high concentration of terrestrially derived organic matter in the study area suggests proximity to the paleoshoreline (Obloh, 1992; Habib *et al.*, 1994; Carvalho *et al.*, 2013; Lorente *et al.*, 2014; Mahmoud *et al.*, 2013; Zarei, 2017; Tabar & Slimani, 2019). Generally, large amounts of phytoclasts particles, as observed in the present study, are deposited by rivers in estuarine and deltaic environments, both of which are closed to shorelines (e.g., Obloh-Ikuenobe *et al.*, 2003). High percentage of phytoclasts show oxidizing conditions and the relative resistance of the lining tissues that are also associated with the proximity of the source area (e.g., Tyson, 1995; Dalseg *et al.*, 2016; Aggarwal *et al.*, 2017). However, palynofacies type I (P-I) and type III (P-III) could be classified as type III kerogen (mainly gas prone, could be related to the high phytoclasts input) as deduced mainly from Tyson (1993) ternary plot (Jaeger, 2013; Tahoun *et al.*, 2013; Makled & Baioumi 2013). The kerogen investigation indicates that particulate organic matter, spore coloration and thermal maturity results obtained from the Enugu outcrop samples is at an immature thermal oil maturity stage (Atta-Peters & Kyorku, 2013; Makled *et al.*, 2013; Ribeiro *et al.*, 2013; Chiaghanam *et al.*, 2014; Atta-Peters *et al.*, 2015).

Conclusion

The Campanian/Maastrichtian Enugu shale outcrop samples are characterized, in order of abundance and diversity, by pteridophyte (spores), angiosperm pollen (e.g., palmae and others), freshwater algae, dinoflagellate cysts, gymnosperms, fungal elements, acritarchs and microforaminiferal wall linings. Using palynomorphs, seven informal palynofloral assemblage zones (Assemblage Zone A to G) were delineated. The assemblage zones were correlated with major cycles of alternating dry and wet climatic conditions in the studied area. On the basis of qualitative and quantitative analyses of the particulate organic matter recovered in the studied samples, four palynofacies assemblages were proposed, and their equivalent kerogen types are identified. The four palynofacies assemblages are: Palynofacies type I (P-I), Palynofacies type II (P-II), Palynofacies type III (P-III) and Palynofacies type IV (P-IV). The paleoenvironmental conditions which prevailed during deposition of the studied sediments were inferred from the integration of palynofacies and the other palynological data. The environments of deposition ranges between fluvial to shallower marine conditions. Spore exine colors and thermal maturity analysis pointed to an organic content that is thermally immature.

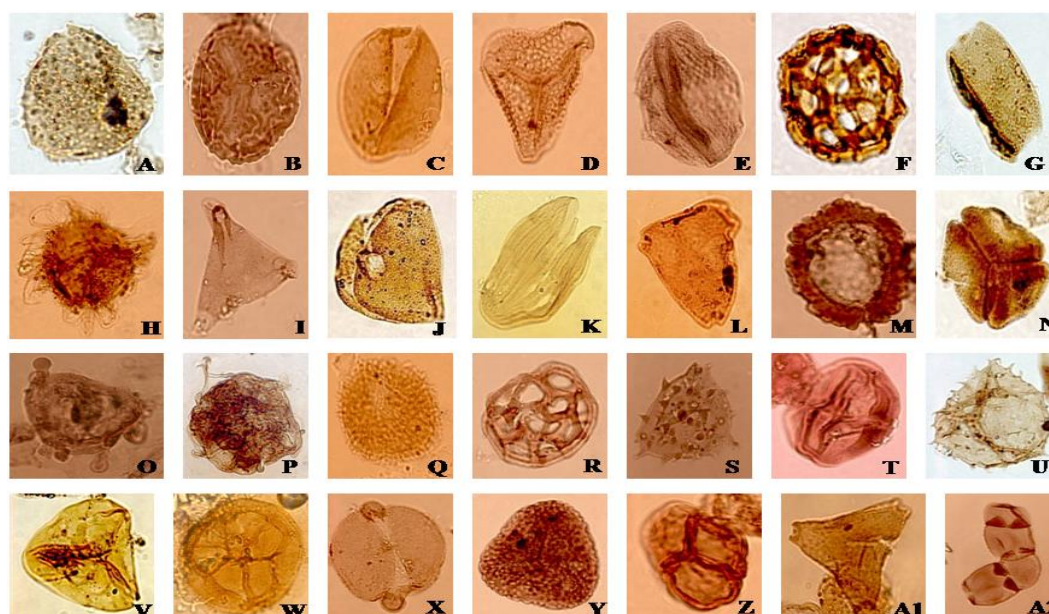


PLATE I: Photomicrographs of some pollen and spores recovered from the Enugu Shale: **A**, *Echitriporites trianguliformis* Van Hoeken-Klinkenberg 1964 (Sample 23 P23/4); **B**, *Rugulatisporites caperatus* Van Hoeken-Klinkenberg 1964 (Sample 10 J38/4); **C**, *Monocolpites marginatus* Van der Hammen 1954 (Sample 11 R45/1); **D**, *Cupaniedites reticularis* Cookson and Pike 1954 (Sample 11 W34/1); **E**, *Gnetaceaepollenites* sp. (Sample 11 F52/2); **F**, *Buttinia andreevi* Boltenhagen 1967 (Sample 12 R57/4); **G**, *Retidiporites magadalenensis* Van der Hammen and Garcia, 1966 (Sample 1 P32/4); **H**, *Ariadnaesporites nigeriensis* Odéboché & Skarby, 1980 (Sample 11 M47/3); **I**, *Aquilapollenites* sp. (Sample 11 R45/1); **J**, *Longapertites vaneendenburgi* Germeraad et al. 1968 (Sample 12 U28/1); **K**, *Ephedripites multicostatus* Brenner, 1963 (Sample 12 F30/0); **L**, *Proteacidites dehaani* Germeraad, Hopping and Muller, 1968 (Sample 23 P23/4); **M**, *Cingulatisporites ornatus* van Hoeken-Klinkenberg, 1964 (Sample 12 F30/0); **N**, *Syncolporites marginatus* Van Hoeken-Klinkenberg, 1964 (Sample 12 R51/2); **O**, *Spinizonocolpites baculatus* Muller et al., 1968 (Sample 8 P28/4); **P**, *Azolla cretacea* Stanley, 1965 (Sample 12 X38/0); **Q**, *Constructipollenites ineffectus* Van Hoeken-Klinkenberg, 1966 (Sample 1 K35/3); **R**, *Buttinea andreevi* Boltenhagen 1967 (Sample 12 R57/4); **S**, *Ariadnaesporites nigeriensis* Odéboché & Skarby, 1980 (Sample 7 Z43/1); **T**, *Psilastephanocolporites* sp. (Sample 23 P23/4); **U**, *Inaperturites cristatus* van Hoeken-Klinkenberg, 1964 (Sample 2 J36/3); **V**, *Cyathidites australis* Couper, 1953, Kar 1990 (Sample 12 P30/2); **W**, *Zlivisporis blanensis* Pacltova 1961 (Sample 19 J53/2); **X**, *Auriculiidites reticulatus* Elsik, 1964 (Sample 23 P23/4); **Y**, *Foveotriletes margariae* Germeraad et al. 1968 (Sample 8 N36/4); **Z**, *Ericipites pachyexinus* Salami 1985 (Sample 2 S44/2); **A1**, *Proteacidites sigalii* Boltenhagen, 1978 (Sample 2 S44/2); **A2**, Fungal spore (Sample 23 P23/4). Bar scale = 20µm

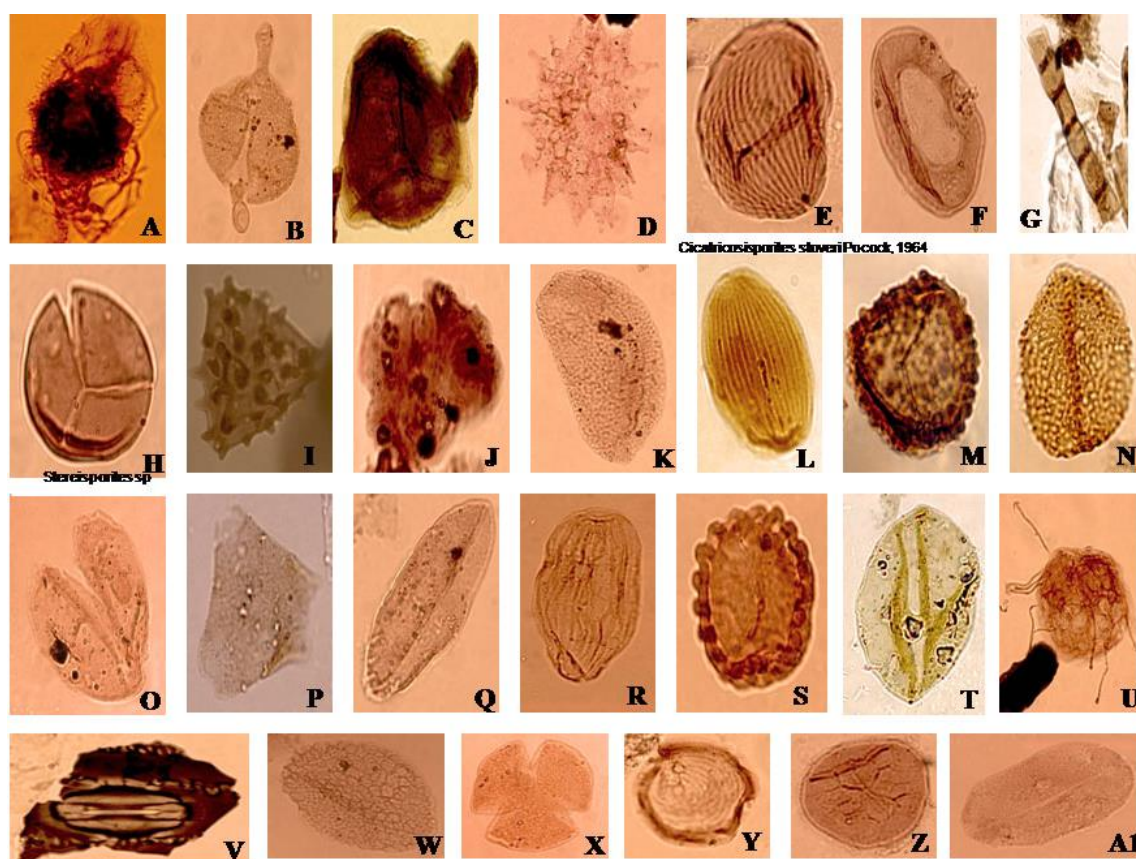


PLATE II: Photomicrographs of some pollen and spores recovered from the Enugu Shale: **A**, *Ariadnaesporites spinosa* (Elsik) Günther and Hills. 1970 (Sample 21 M34/4); **B**, *Auriculipollenites* sp.

(Sample 3 R36/3); **C**, *Deltoidospora* sp. (Sample10 C41/2); **D**, *Pediastrum* sp. (Sample10 C41/2); **E**, *Cicatricosisporites sloveri* Pocock 1964 (Sample 3 R42/2); **F**, *Milfordia jardinei* Erdtman 1960 (Sample 9 P56/1); **G**, Fungal spore (Sample 6 G48/1); **H**, *Stereisporites* sp., (Sample 19 T27/2); **I**, *Echitriporites longispinosus* (Jardiné and Margloire, 1965) Shrank, 1994 (Sample 5 N40/2); **J**, *Botryococcus* sp. (Sample 2 N39/0); **K**, *Longapertites marginatus* Van Hoeken-Klinkenberg, 1964 (Sample 5 Q36/1); **L**, *Ephedripites* sp. (Sample 5 Y38/2); **M**, *Distaverrusporites simplex* Muller, 1968 (Sample 19 W32/4); **N**, *Longapertites reticulatus* Salami 1990 (Sample 3 U29/2); **O**, *Monocolpopollenites sphaeroidites* Jardiné and Margloire, 1965 (Sample 19 Y25/3); **P**, *Cretaceaeporites polygonalis* (Jardiné and Margloire) HERNGREEN, 1974 (Sample 2 K59/4); **Q**, *Longapertites microfoveolatus* (Sample 9 C41/2); **R**, *Ephedripites* (Sample10 C41/2); **S**, *Tubistephanocolporites cylindricus* Salami 1985 (Sample 7 U53/3); **T**, *Sapotaceoidaepollentites* sp. (Sample 13 F281/2); **U**, *Azolla cretacea* Stanley, 1965 (Sample 9 L29/3); **V**, Charred gramineae cuticle (Sample 9 M46/3); **W**, *Liliacidites* cf *nigeriensis* (Van Hoeken-Klinkenberg 1966) Salami 1985 (Sample 9 K29/3); **X** *Retitricolporites* sp. (Sample 8 M37/4); **Y**, *Concentricytes circulus* (Sample 9 M38/1); **Z**, *Tubistephanocolporites cylindricus* Salami 1985 (Sample10 C41/2); **A1**, *Longapertites microfoveolatus* (Sample 8 K30/2). Bar scale = 20µm

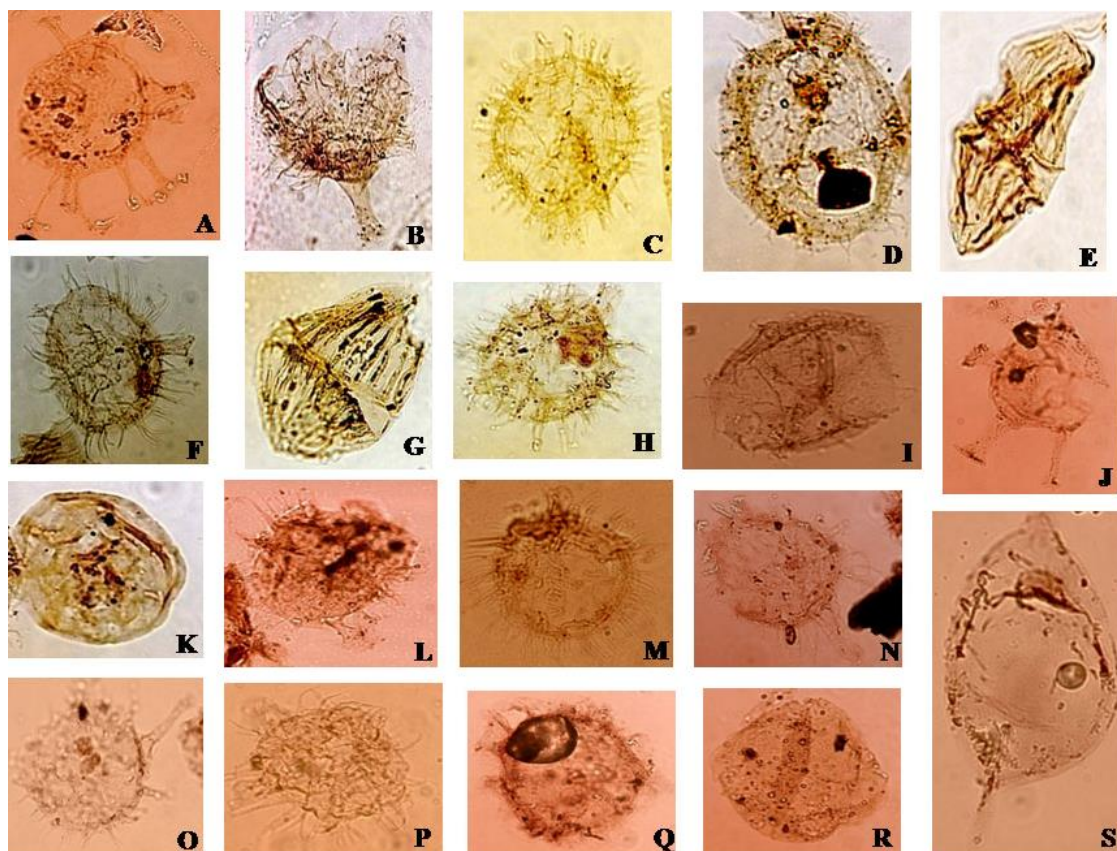


PLATE III: Photomicrographs of some dinoflagellate cysts recovered from the Enugu Shale: **A**, *Florentinia* cf *lacinata* Davey and Verdier 1973 (Sample 4 K51/4); **B**, *Dinogymnium nelsoense* (Sample 12 Q52/2); **C**, *Palaeocystodinium gabonensis* (Sample 21 Z35/2); **D**, *Florentinia resex* (Deflandre & Cookson 1955) (Sample 9 Q50/4); **E**, *Thalassiphora pelagica* (Eisenack, 1954) Eisenack & Gocht, 1960 (Sample 2 N43/0); **F**, *Achomosphaera* sp. (Sample 15 L45/4); **G**, *Coronifera oceanica* Cookson and Eisenack, 1958 (Sample 17 Y46/0); **H**, *Cyclonephelium* cf *vannophorum* (Davey, 1969) (Sample 17 S34/3); **I**, *Andalusiella* cf *polymorpha* (Malloy) Lentin & G.L. Williams 1977 (Sample 2 Y26/0); **J**, *Exochosphaeridium bifidum* R.J. Davey, 1966 (Clarke and Verdier, 1967 (Sample 1 T28/3); **K**, *Operculodinium*

sp. (Sample 14 R42/4); **L**, *Coronifera* sp. (Sample 16 Y55/4); **M**, *Spiniferites* sp. (Sample 12 S36/2); **N**, *Coronifera oceanica* Cookson and Eisenack, 1958 (Sample 12 R57/2); **O**, *Hystrichosphaeridium tubiferum* (Ehrenberg, 1838) Deflandre, 1937; emend. Davey & Williams, 1966 (Sample 15 R46/2); **P**, *Pervosphaeridium pseudohystrichodinium* (Deflandre 1937b) Yun, 1981 (Sample 16 W49/2); **Q**, *Cleistosphaeridium* sp. (Sample 14 S31/2); **R**, *Florentinia* sp. (Sample 14 Y48/4); **S**, *Senegalinium* sp. (Sample 18 S47/3).

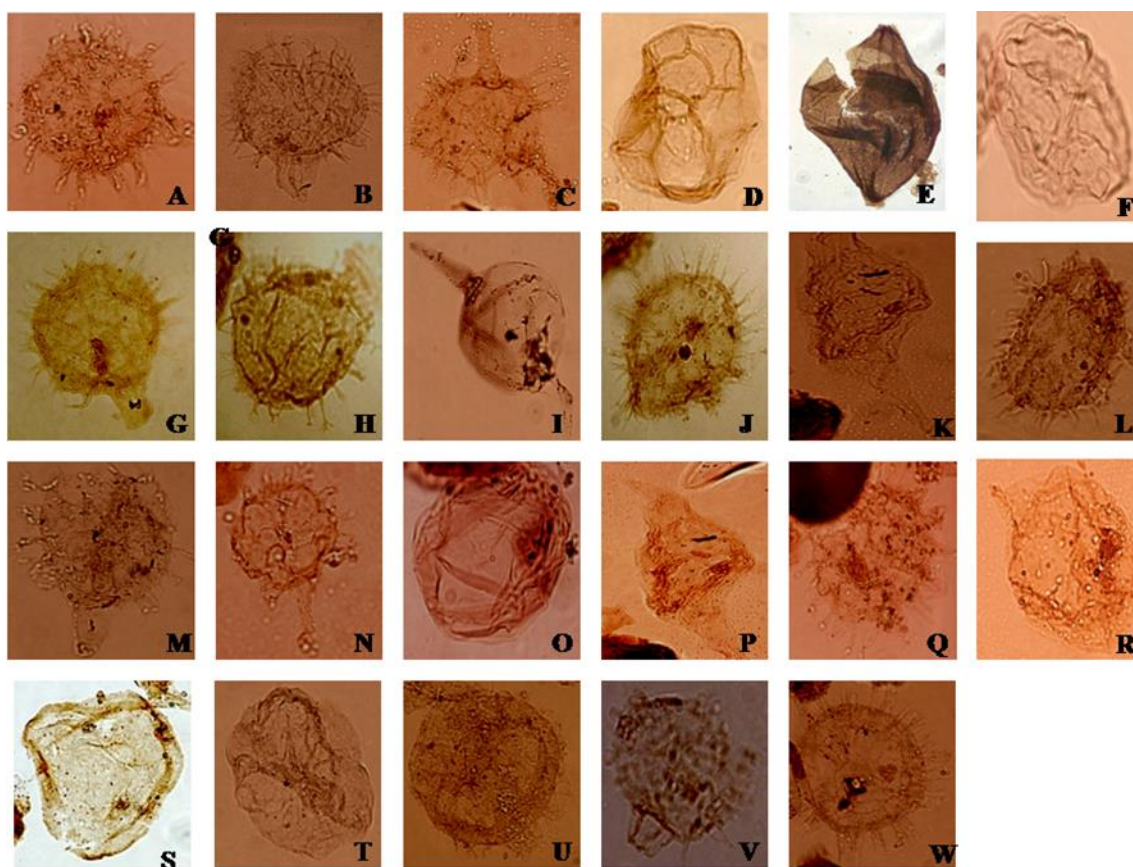


PLATE IV: Photomicrographs of some dinoflagellate cysts recovered from the Enugu Shale: **A**, *Florentinia* sp. (Sample 4 K51/4); **B**, *Exochosphaeridium bifidum* R.J.Davey, 1966 (Clarke and Verdier, 1967 (Sample 12 N44/4); **C**, *Pterodinium* sp. (Sample 21 X52/3); **D**, *Diphyes colligerum* (Deflandre & Cookson 1955) Cookson 1965a emend. Goodman & Witmer 1985 (Sample 22 M33/4); **E**, *Oligosphaeridium complex* (White) (Davey and Williams, 1966) (Sample 11A S47/1); **F**, Indeterminate dinocyst (Sample 7 K41/2); **G**, *Areoligera* sp. (Sample 3 X37/40); **H**, *Dinogymnium acuminatum* Evitt *et al.*, 1967 (Sample 12 W30/2); **I**, *Phelodinium* sp. (Sample 18 F45/4); **J**, *Hystrichokolpoma* sp. (Sample 13 T28/3); **K**, *Palaeocystodinium australinum* (Cookson, 1965) Lentin & Williams, 1976 (Sample 21 T41/0); **L**, *Canningia* sp. (Sample 3 V56/0); **M**, *Cleistosphaeridium* sp. (Sample 13 V56/0); **N**, *Adnatosphaeridium* cf. *buccinum* Hultberg 1985 (Sample 3 J30/2); **O**, *Spiniferites* sp. (Sample 2 L53/2); **P**, *Florentinia ferox* (Sample 13 V56/0); **Q**, *Exochosphaeridium bifidum* (Sample 9 P48/3); **R**, *Cleistosphaeridium* sp. (Sample 7 M27/1); **S**, *Trichodinium* sp. (Sample 6 N39/3); **T**, *Selenopemphix* sp. (Sample 5 W35/4); **U**, *Diphyes colligerum* (Deflandre & Cookson 1955) Cookson 1965a emend. Goodman & Witmer 1985 (Sample 6 P52/4); **V**, *Cribroperidinium* sp. (Sample 8 R35/4); **W**, *Achomosphaera* sp. (Sample 13 H41/2). Bar scale =20µm

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