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Micropaleontology and inorganic geochemical studies of the Campanian-maastricthian shale sediments in the Anambra and southern Bida basins Nigeria: Implication for provenance and paleoenvironment

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ABSTRACT

The Micropaleontology and Inorganic Geochemical analysis were carried out on the shales of Enugu and Patti Formations (Anambra and Southern Bida Basins) deposited during the Campanian-Maastrichtian time interval to evaluate the connectivity of the paleo-sea depositional environment and sediments source. The method of analysis employed includes field sampling of two exposed sections of the formations and laboratories analyses which includes foraminiferal studies, Xray Fluorescence spectrometry (XRF) and Xray diffraction analysis (XRD). 30 selected shale samples were used for these analyses. The lithologic sections presented shows similarities in the reconstructed visible layers of sediment by the exposed sections in the two formations. The foraminiferal analysis revealed abundance of Benthic species depicting a shallow marine environment for the deposition of the sediments. The Benthic species recovered includes, *Ammobaculite coprolithiformis, Ammobaculites exiguus, Ammobaculites strathearnensis, Ammobaculoides parvus, Ammomarginulina* cf. *A. curvature, Bolivina afra, Eponides* sp, *Haplophragmoides bauchensis, Haplophragmoides bausa, Haplophragmoides saheliense, Lenticulina cultrataspecies, Miliammina petila, Nonionella robusta, Praebulimina bantu, Textularia hockleyensis* and *Textularia parvula*. The XRF analysis results of the selected shale samples of the two formations revealed SiO_2 as the dominant oxide (Average Enugu-87.06 wt % and Patti-62.42 wt %). The Al_2O_3/TiO_2 ratios in the two formations suggests that source rocks of these formations are likely to be transitioning from mafic to intermediate rock composition. Plot of SiO_2/Zr display positive correlation in the shale of the Patti Formation to indicate lack of biogenic silica while showing negative correlation in that of Enugu Formation suggesting favorable environment for biotic life. Also, the plots of TiO_2/Zr and TiO_2/Al_2O_3 described both formations as intermediate rock composition for provenance. The plot of V/Ni revealed both formations as marine anoxic paleoenvironments. The Xray diffraction analysis reveals abundance of quartz and small amount of accessory minerals like goethite, kaolinite, smectite, mica, anatase, siderite and calcite.

1. Introduction

Over time, scientists have dedicated significant efforts to investigate the ancient settings in which sedimentary rocks are formed in various sedimentary basins. Sediment deposition takes place under distinct physical and chemical circumstances that can differ between different geological eras. The examination of these deposition environments can be carried out at both small and large geographical levels. Various initiatives have been undertaken to evaluate the potential of Nigerian sedimentary basins (Adeleye, 1989; Obi, 2000; Ogbahon and Olujinmi, 2019; Olabode, 2016; Tijani et al., 2010). The lack of comprehensive records regarding shifts in sea levels resulting from sediment buildup during the **Campanian-Maastrichtian** period, particularly in Nigeria, indicates a gap in our understanding. It is crucial to evaluate how far the ancient seawater reached in the region while the sediments were being deposited in the mentioned time interval. The aim of this study is to evaluate how the ancient seawater was interconnected within the basins and how it relates to the environments where sediment deposition occurred. The two basins of interest include the **Anambra Basin** and the **Bida Basin** (Fig. 1). Sediments of these basins were analyzed for their elemental composition, mineralogy, and foraminiferal analysis to compare their characteristics during deposition in the Geologic time

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interval. The basins are both inland basins and are in different parts of the country i.e., the North-central part and South-Eastern part of Nigeria (Fig. 1).

2. Geology and stratigraphic settings

The origin of Anambra Basin is associated with the mid-Santonian compressional folds, faults, and uplift in several places in the Benue trough (Obaje, 2009). Anambra basin is a related structure that developed after the compression phase (Akande and Erdtmann, 1998). It serves as a depositional center during the Cretaceous period, where sediments ranging from the Campanian to the Tertiary era were deposited (Nwajide, 1990; Obi, 2000). Among these, approximately 3000 to 3500 m of sediment accumulated during the late Cretaceous period, specifically from the Upper Campanian to the Maastrichtian (Uzoegbu and Ikwuagwu, 2016). The Anambra Basin stratigraphy (Fig. 2) started to accumulate sediments with the shales of the Enugu and Nkporo Formations, which were formed in marine and coastal environments during the Campanian-Maastrichtian period and were covered by the coal-bearing layers of the Mamu Formation, then overlayed by Ajali sandstone and Nsukka Formation which was succeeded by Imo shale and finally capped by Ameki group (Adeleye, 1989; Obaje, 2009; Obi, 2000; Reyment, 1965)

The prevailing viewpoint regarding the origin of the Bida Basin suggests that it was formed by the deposition of upper Cretaceous sediments. The evolution of the Bida Basin is also like that of the Anambra Basin formed during the Santonian orogenic movements of southeastern Nigeria and the Benue valley, nearby. Many people consider it as the part of the Anambra Basin that extends to the northwest, and both basins were important places of sediment accumulation during the Late Cretaceous period when the third major sea level rise occurred in southern Nigeria. The Bida Basin sediments consist mainly of clastic materials and were laid down within a rift basin that emerged due to the gradual separation of the South America and Africa tectonic plates (Agyingi, 1991; Kennedy, 1965; King, 1950). Conclusions drawn from relevant research (Kogbe et al., 1983; Ojo and Ajakaiye, 1989) indicated prominent faults oriented in the NW/SE direction. Additionally, there was evidence of a central positive anomaly at depth, surrounded by negative anomalies within the basin. These findings lent support to the concept of a rift, or continental separation, theory. In contrast, (Adeleye, 1974; Whiteman, 1982) put forth the notion of a straightforward post-Santonian cratonic sag as an alternative explanation. In the southern Bida Basin, three identifiable stratigraphic horizons within the Campanian-Maastrichtian period have been mapped out and explained (Fig. 2). These horizons from the oldest to youngest consist of the Lokoja Formation, Patti Formation and Agbaja Formation(Jones, 1955; Obaje, 2009; Ojo and Akande, 2003)

3. Materials and methods

The methods used in this research work involves Fieldwork for lithological descriptions, sample collection and laboratory analyses. Samples used in this study are outcrop samples taken from roadside exposure of the Enugu Formation at Latitude N7°24′58″ N and Longitude 6°55′12″ E exposed at Ojodu town, Ajaokuta-Ayangba express road and Patti Formation at Latitude 8°18′5″ N and Longitude 6°51′29″ E exposed at Ahoko town, Lokoja-Abuja express road (Fig. 1). The samples were subjected to microfossil, inorganic geochemical and XRD analyses.

Microfossil investigation involves foraminiferal analysis and it takes place at the micropaleontological laboratory of the Geology

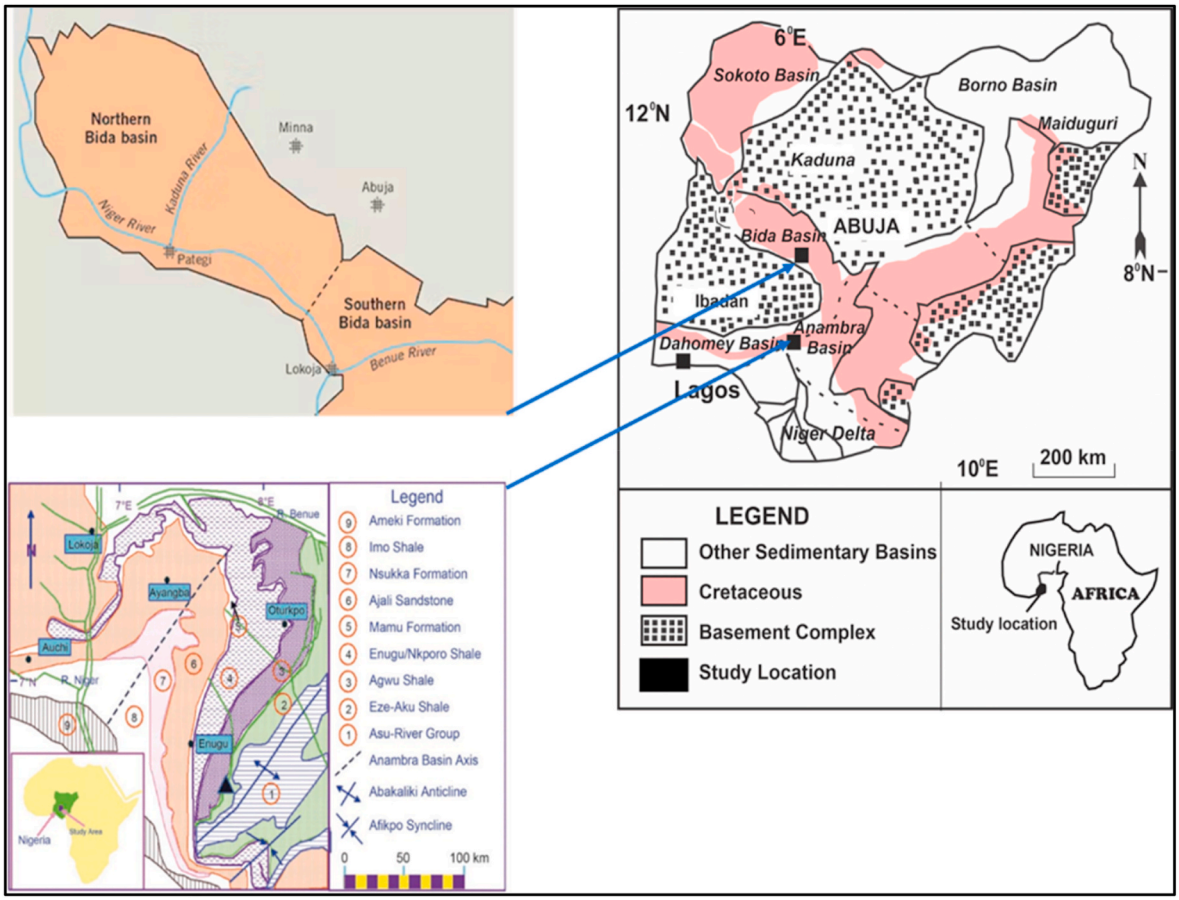


Fig. 1. Geological map of the study locations in southern Bida Basin and Anambra Basin (modified after Obaje et al., 2011; Nton and Bankole, 2013).

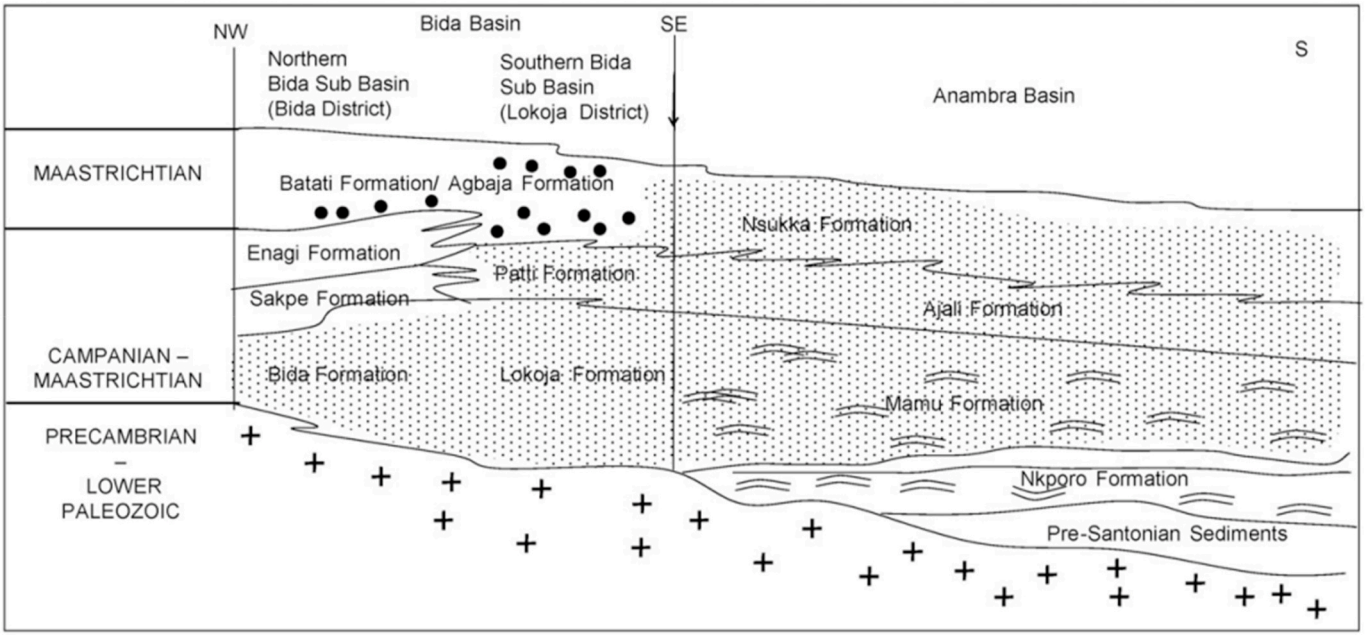


Fig. 2. Regional stratigraphic settings of the Northwest Anambra Basin and Bida Basin (After Akande et al., 2005).

Department, University of Ilorin. 40g each of the 30 selected shale samples from the two basins was put in a labeled bowl, concentrated hydrogen peroxide mixed with water in a ratio of 1:3 was poured into each sample. The samples were left overnight to dissolve and after which it was washed under running water using 63 μm sieve size, the residue retained was poured onto a plate and dried after which it was poured into a bottle for storage. Using relevant literature, such as (Bolli, 1957, 1966; Bolli and Saunders, 1985), foraminiferal species were picked and identified from the residue using Zeiss V8 stereomicroscope with 0.63x auxiliary lens and the photomicrograph were taken by the use of Zeiss EVO 15 scanning electron microscope (SEM) in laboratory of the Goethe University Frankfurt, Germany. All the remaining residue and foraminifera species recovered are kept in paleontological laboratory of the department of Geology and mineral science, University of Ilorin, Nigeria.

10 Selected shale samples were also pulverized and subjected to x-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses at the geology laboratory of Free State University, South Africa. The process of XRF involved grinding the samples using an iron milling machine. The loss of mass due to ignition was measured by subjecting the sample to a temperature of 950 °C. All the elements present were examined using a fused bead method, except for Na_2O which was analyzed using a pressed pellet technique. The analytical instrument used was a Rigaku-Primus IV with a Rhodium tube, and the software utilized for this machine was ZXS.

XRD examination was carried out using a Panalytical Empyrean

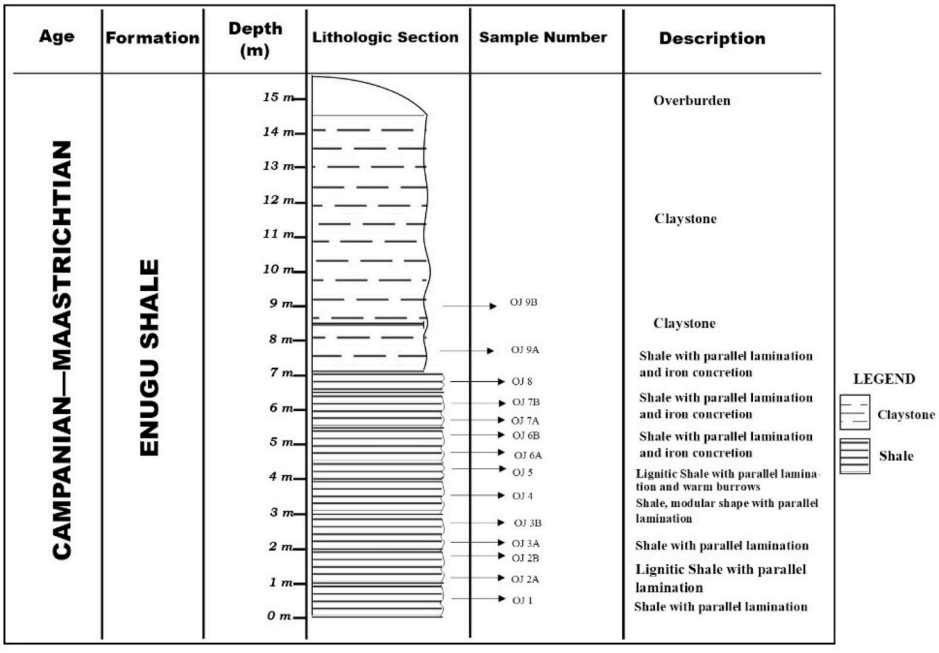


Fig. 3. Lithostratigraphic description of Enugu Formation exposed at Ojodu village.

instrument equipped with a copper-anode X-ray tube. The Highscore software was employed for interpreting the data, in conjunction with the ICDD-PDF2 2021 database. The outcomes represent an interpretation and possess a semi-quantitative nature.

4. Results and interpretation

4.1. Lithological description

4.1.1. Enugu Formation

The area under study is the exposed part of the Enugu formation of Anambra Basin at Ojodu town Ajaokuta-Ayangba express road. The section is about 14.6m high and is a deposition of thick sequence of marine shale and claystone deposited during the Maastrichtian (Fig. 3). The basal part of the outcrop contains alternate beds of laminated dark shale with massive bedded and nodular beds (Fig. 5). The middle part comprises parallel laminated grey shale beds with lignitic coaly particles. The upper part consists of claystone bed and lateritic overburden. There is presence of worm burrows within the shale beds.

4.1.2. Patti Formation

The Patti formation of Southern Bida Basin is exposed at Ahoko village, Lokoja-Abuja express road (Fig. 4). It is about 8.1 m thick and also a similar deposition to Enugu Formation comprising of sequence of dark laminated shale beds alternating with massive bedded, nodular shale bed with evidence of trace fossils at the base (Fig. 5**).** Within the shale beds are thin intercalation of ironstone beds. The upper part comprises siltstone and claystone beds.

4.2. Foraminiferal studies

The presence of foraminifera in shale deposits from the Campanian-Maastrichtian within the Enugu and Patti Formations provided valuable information about the past ecological conditions and paleoenvironment. This information is beneficial for understanding ancient environmental reconstruction. The abundance of planktonic and benthic Foraminifera species differs from each other in terms of their diversities. Several foraminiferal species, mostly benthic and very few planktonic species were recovered from the residue of the two basins under study (Table 1). Most of the species are agglutinated (arenaceous) and only very few are calcareous. The reason for the agglutinated forms is because of post depositional changes that has acted upon these forms over the years and as a result of their closeness to the continent where temperature, acid action and oxygen actions has acted upon the shells over long period of time after deposition.

Several benthic foraminifera species recovered includes *Ammobaculite coprolithiformis, Ammobaculites exiguus, Ammobaculites strathearnensis, Ammobaculoides parvus, Ammomarginulina* cf. *A. curvature, Eponides* sp, *Haplophragmoides bauchensis, Haplophragmoides bausa, Haplophragmoides* saheliense, *Lenticulina cultrataspecies, Miliammina petila, Nonionella robusta, Praebulimina bantu, Portatrochammina* sp, *Textularia hockleyensis, Textularia parvula*. While the Planktic species recovered includes *Ammobaculites* cf. *A. pindigensis, Trochaminna taylorana, Haplophragmoides pettersi* sp. (Figs. 6 and 7)

4.3. Geochemistry

The geochemical data for both major oxides and trace elements of the analyzed sediments from the two basins, along with their statistical associations are provided in Tables 2 and 3. The results presented for the Enugu Formation reveals SiO_2 ranges from 82.55 wt % - 91.43 wt %, TiO_2 ranges from 0.68 wt %-1.08 wt %, Al_2O_3 ranges from 5.5 wt % - 8.57 wt %, Fe_2O_3 ranges from 0.49 wt % - 1.78 wt %, and Na_2O, K_2O, CaO, MnO, P_2O_5, and MgO exhibit generally lesser presence (Table 1). Subsequently the results presented for the Patti Formation reveals SiO_2 ranges from 56.83 wt % - 69.16 wt %, TiO_2 ranges from 1.91 wt, %-2.69 wt %, Al_2O_3 ranges from 15.02 wt % - 22.03 wt %, Fe_2O_3 ranges from 1.07 wt % - 3.29 wt %, These findings were also cross-referenced with the Post-Archean Australian Shale (PAAS) values according to (Pettijohn, 1975; Taylor and McLennan, 1985). The levels of MgO, MnO, and Na_2O in both samples were quite minimal, adding up to less than 2 % in total. The quantities of MgO and CaO were also not substantial. Therefore, the samples did not demonstrate the presence of carbonates or dolomitization. The silica contents are high in the shales of Enugu Formation with an average value of 87.06 % when compare to that of Patti formation with average of 62.42 %. Whereas the alumina (Al_2O_3) contents in Patti formation is higher in shales of Patti formation with average value of 20.45 %. High levels of alumina are frequently

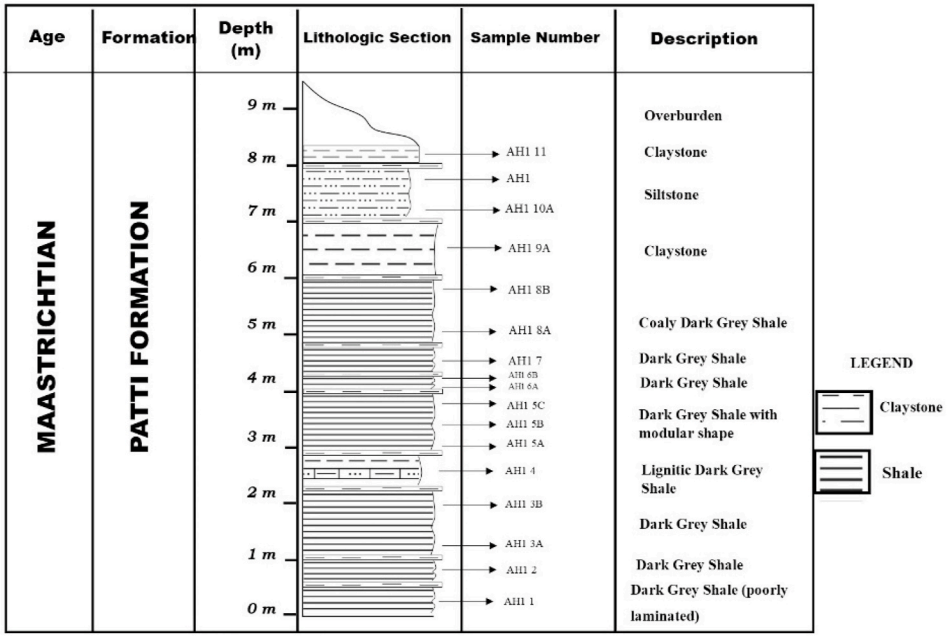


Fig. 4. Lithostratigraphic description of Patti Formation exposed at Ahoko village.

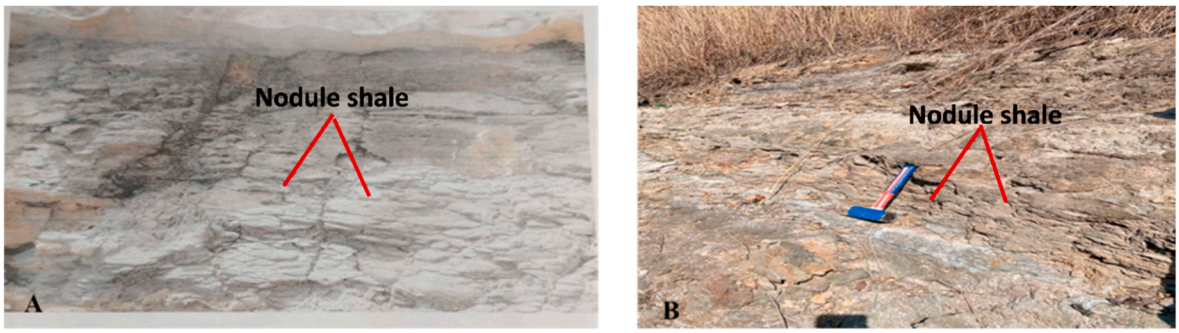


Fig. 5. Field photograph showing nodular shale occurrence in shales of Patti Formation and Enugu Formation showing similarities (A-Patti Formation, B-Enugu Formation).

observed in sediment types characterized by clay and clay-rich compositions (Adeoye et al., 2020). It was also observed that the Iron (Fe_2O_3) in the shale of Patti Formation is higher with an average value of 2.27% compared to value of Enugu Formation with values of 0.89 % (Table 2). This increase is from the ironstone present within the Shale of the Patti Formation. Additionally, Table 3 provides the concentrations, variations, and ranges of trace elements in the studied sediments, alongside corresponding standard values for comparison. A total number of 17 trace elements were recorded including Sc, V, Cr, Co, Ni, Cu, Yn, As, Rb, Sr, Y, Zr, Nb, Ba, Pb, Th and U. These Trace elements are usually derived from terrestrial remains of weathered rocks, Planktons or mineral-rich waters from hydrothermal sources (Leventhal, 1998; Tribovillard et al., 2006).The average value of Vanadium in the shale of Enugu Formation is 56 ppm and Patti Formation has an average value of 122.6 ppm. These values when compared to the publish values of Post – archean Australian shale (PAAS) is slightly lower in Patti Formation but very low in Enugu Formation (Table 3). The ratios of the trace elements were also compared to the post-archean Australia shale (PAAS), the values of V/Ni, Ni/Co, Th/Co and Th/Sc are all lower than the PAAS values (Table 4).

4.4. Minerology

The X-ray diffraction patterns (XRD) of the entire rock samples from the Enugu Formation's shale indicated the presence of various minerals, including kaolinite, quartz, Goethite, jarosite, smectite, mica Anatase, K-feldspar, Siderite, and Calcite (Table 5). Similarly, the XRD result for the Patti Formation's shale also displayed the presence of kaolinite, quartz, Goethite, jarosite, smectite, mica Anatase, K-feldspar, and Siderite. In both formations, Quartz was the most abundant mineral, and Kaolinite emerged as the predominant clay mineral. The characteristic diffraction patterns of the shales showcased Quartz as the prevalent mineral (making up 66% of the composition), while minerals like Kaolinite, Jarosite, Smectite and Siderite were present in smaller quantities as illustrated in (Fig. 8).

5. Discussion

5.1. Provenance

Stationary elements like such as Al, Ti, Th, Sc, Co, Nb, Ta, Y, and Zr offer significant insights into the origin, geological processes, and erosion within the area of origin (Gu et al., 2002; Hossain et al., 2010; Jorge et al., 2013; McLennan et al., 1993). The utilization of these stationary components in determining origin relies on the presumption that these elements experience minimal geochemical differentiation during sedimentary activities. As a result, they are integrated into sedimentary archives in a manner that corresponds directly to their concentrations in the original region (McLennan et al., 1990; Singh, 2009). The primary source of silica originates predominantly from terrestrial detrital quartz, with infrequent contributions from biogenic origins. Silica is frequently found in conjunction with clay-rich sediments like clay minerals (Pettijohn, 1975). The most abundance oxides recovered from the shales of Enugu and Patti Formations is Silica (SiO_2). This signify that terrestrial materials are part of the predominate source of these sediment.

The chemical characteristics of clastic sediments have been utilized to identify their origin properties (Armstrong-Altrin et al., 2004; Condie et al., 1992; Cullers, 1995). For most clastic rocks, the ratio of Al_2O_3 to TiO_2 is a useful indicator of source rock composition. This ratio ranges from 3 to 8 wt % for mafic igneous rocks, from 8 to 21 wt% for intermediate rocks, and from 21 to 70 wt % for felsic igneous rocks (Hayashi et al., 1997). In the Patti Formation, the Al_2O_3/TiO_2 ratio varies from 7.62 wt % to 10.98 wt %, and in the Enugu Formation, it ranges from 7.53 wt % to 9.07 wt %. Consequently, based on the Al_2O_3/TiO_2 ratio, this study suggests that the source rocks for both formations, are likely to be transitioning from mafic to intermediate rock compositions.

The graph illustrating SiO_2 against Zr in Fig. 9 displays a positive relationship in shales of the Patti Formation, implying the lack of biogenic silica presence. This is due to the fact that Zr is commonly linked with the heavy mineral zircon, which serves as an indicator for terrestrial siliciclastic substances (Wright et al., 2010). This observation could additionally indicate a hostile environment for life, possibly due to insufficient oxygen levels. On the other hand, the correlation between SiO_2 and Zr is negative in shales of Enugu Formation, suggesting the existence of certain biogenic silica content. The plot of TiO_2 against Zircon and TiO_2 against Al_2O_3 (Ofili et., al 2022), described both the shale of Patti Formation and Shale of Enugu Formation as intermediate rock composition (Figs. 10 and 11).

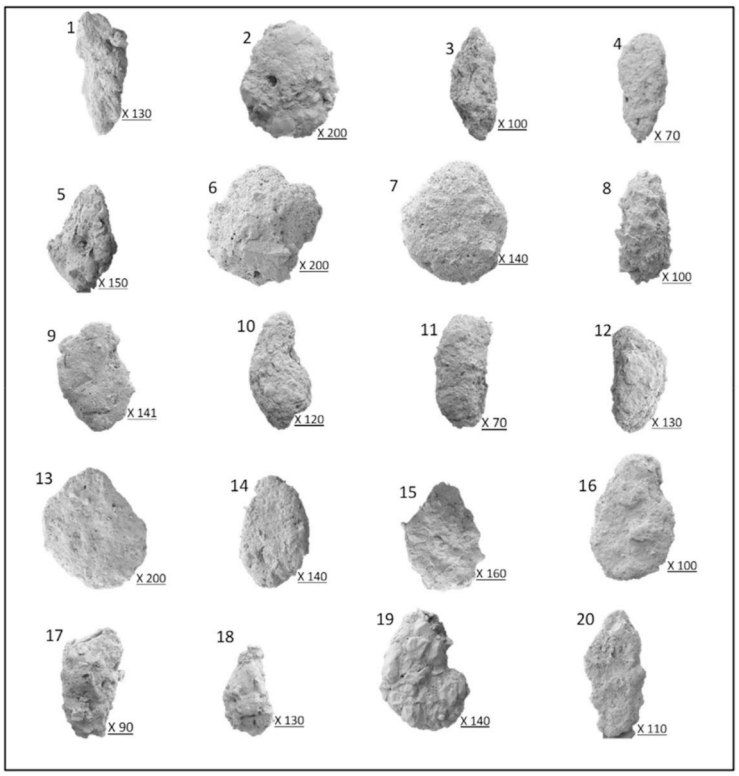
The mineral composition in both studied locations depict same or similar depositional process in both basins. The high presence of Quartz could be interpreted that the depositional environment has high influence of terrestrial detrital sediments.

5.2. Paleoenvironment

By examining benthic foraminiferal assemblages, which are highly dependent on environmental factors like the availability of nutrients, the oxygenation level in the seafloor water, paleo – salinity, temperature and paleo-bathymetry we were able to deduce the paleoenvironmental conditions of the Campanian-Maastrichtian sediments within the basins. In these basins, the diversity of benthonic foraminifera is minimal but a significant proportion of *Haplophragmoides, Ammobaculites* and *Ammotium* suggests brackish-water (Murray, 1991). A near-shore environment was implied by the presence of agglutinated benthonic foraminifera in the basins. The abundance of benthic species with respect to low representation of planktic form also suggest a shallow marine environment for the deposition of both Enugu and Patti shales. Salinity is one of the primary factors influencing the distribution of marine microfossils, such as foraminifera (Hamza et al., 2011), Planktonic species have low tolerance to low salinity and benthic foraminifera species are known to

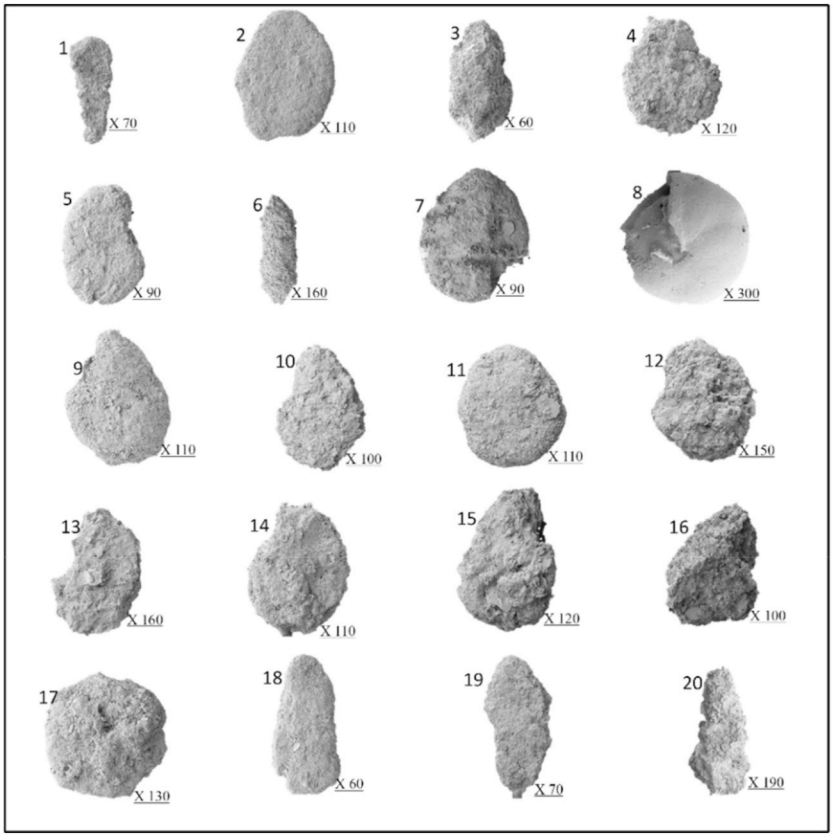
Table 1
Distribution of Benthic foraminiferal picked from the selected basins.

S/N	*Ammobaculitecoprolithiformis*	*Ammobaculitescf.A. pindigensis*	*Ammobaculitesexiguus*	*Ammobaculitesstrathearnensis*	*Ammobaculoidesparvus*	*Ammomarginulinacf.A. curvatura*	*Ammotiumlerneusis*	*Ammotiumwalium*	*Bolivinaafra*	*Elphidellaafricana*	*EponidesSp*	*Haplophragmoidesbauchensis*	*Haplophragmoidesbausa*	*HaplophragmoidesspettersiSp*	*Haplophragmoidessaheliense*	*HaplophragmoidesSp.*	*Lenticulinacultrata*	*Miliamminapetila*	*Miliamminapindigenesis*	*Nonionellarobusta*	*PortatrochamminaSp*	*Praebuliminabantu*	*Praebuliminabantu*	*Textulariahockleyensis*	*Textulariaparvula*	*TextulariopsisDukamajima*	*Trochaminataylorana*
OJ 1	1	1			1		2		1	1		2		1	1		1		1	1		1	1		1	1	1
OJ 2A		1	1		3		2	2									1	1					1				
OJ 2B		1						2	1					2			1		1	2	1	1			1		1
OJ 3A	1		1		1	1	1		1				2	2			1							1			
OJ 3B				1	3		1			1		2	2	2	1			1	1	2	1		1			1	
OJ 4			1																					1			
OJ 5	1	1	3	5	1	3	1	1			8	3	2		1	1	3		1	1	4			3	2		
OJ 6A	4				2			3							2						4	2	2		2		
OJ 6B	1	4	2	3						1			5	8				4	3	1				1			3
OJ 7A			2		1		4	1	1	1		1	1	3	1		2		2	3	3	2					
OJ 7B	1	1		3			1						1					1		5	1			4	2		
OJ 8	1		1		3	1			1			1						2	2	1	1	2		1		2	
AH 1				1	1				1		2			1	1	1	2	1				1	1	1			
AH 2		1	1	1	4														1						2		
AH 3A			1	1		1	1	1									1						1				
AH 4	1				9		1	2	1			1			1	1		3	1	1				1			1
AH 5A		2	3	4				2										3	1				1		5		
AH 5B	2				3		1					1	1	1		1	1		6		3	1					
AH 6		3	6	4		1																		1	5		2
AH 7					1				1				5			1			1		11	1	1			2	
AH 8A			2	1	1			1					3							1							
AH 8B	2			1	2				1				1		1		1		1	2	1	1	1	1			1
Total	12	15	24	25	36	7	15	15	9	4	10	11	23	20	9	5	14	16	22	20	30	12	10	15	20	6	9



1.*Ammobaculoides parvus sp.*
2.*Haplophragmoides bauchensis*
3.*Miliammina pindigensis*
4.*Textulariopsis dukamajina*
5. *Ammotium leruensis*
6.*Haplophragmoides saheliense.*
7.*Praebulimina bantu*
8. *Trochaminna taylorana*
9. *Haplophragmoides pettersi sp*
10.*Ammomarginulina cf. A. curvatura*
11. *Ammobaculites exiguu*
12. *Ammobaculites cf. A. pindigensis*
13. *Lenticulina cultrate*
14.*Ammobaculoides parvus*
15.*Ammotium nwalium*
16. *Nonionella robusta*
17. *Miliammina petila*
18.*Textularia parvula*
19.*Haplophragmoides Bausa*
20. *Textularia hockleyensis*

Fig. 6. Foraminiferal species recovered from Enugu Formation.



1. *Textulariopsis dukamajina*
2. *Praebulimina bantu*
3.*Ammobaculites cf. A. pindigensis*
4.*Nonionella robusta plummer?*
5.*Haplophragmoides pettersi sp*
6.*Ammobaculites exiguus*
7.*Haplophragmoides Sp.*
8. *Eponides Sp.*
9.*Lenticulina cultrate*
10.*Trochaminna taylorana*
11.*Haplophragmoides bauchensis*
12.*? Portatrochammina sp*
13.*Haplophragmoides bausa*
14.*Elphidiella Africana*
15.*Ammomarginulina cf. A. curvature*
16.*Miliammina pindigensis*
17.*Haplophragmoides saheliense*
18.*Textularia parvula*
19.*Ammotium leruensis*
20.*Ammobaculoides parvus sp.*

Fig. 7. Foraminiferal species recovered from Patti Formation.

Table 2
Major oxides of Campanian-Maastrichtian Shale sediments exposed at Ahoko and Ojodu Town.

Major element (wt%)	Patti Formation					Enugu Formation							
	AH1	AH3A	AH4	AH5C	AH8B	OJ1	OJ2	OJ3	OJ4	OJ5	Aver age		PAAS
											Patti	Enugu	
SiO_2	58.02	56.83	61.7	69.16	66.39	87.21	91.43	90.25	83.85	82.55	62.42	87.06	62.40
TiO_2	2.69	2.25	2.06	1.91	2.02	1.08	0.73	0.68	1.02	1.2	2.19	0.94	0.99
$Al2O_3$	20.49	24.7	22.03	15.02	20.03	8.31	5.5	6.17	8.44	8.57	20.45	7.4	18.78
Fe_2O_3	3.29	2.13	1.87	2.97	1.07	0.7	0.57	0.49	0.91	1.78	2.27	0.89	7.18
MgO	0.53	0.26	0.21	0.14	0.6	0.06	0.17	0.09	0.1	0.13	0.35	0.11	2.19
MnO	0.08	0.04	0.02	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.04	0.01	–
CaO	0.23	0.12	0.08	0.07	0.09	0.05	0.05	0.05	0.05	0.05	0.12	0.05	1.29
Na_2O	0.02	0.02	0.02	0.02	0.03	0.01	0.01	0.01	0.01	0.01	0.02	0.01	1.19
K_2O	0.93	1.06	0.97	1.37	1.25	0.55	0.5	0.53	0.46	0.5	1.12	0.51	3.68
P_2O_5	0.19	0.17	0.12	0.07	0.1	0.03	0.02	0.02	0.03	0.05	0.13	0.03	0.16
LOI	13.49	13.08	9.4	9.64	7.43	1.68	2.98	2.87	4.76	5.2	–	–	–
Total	99.96	100.66	98.48	100.41	99.03	99.7	101.96	101.18	99.64	100.04	–	–	–

Table 3
Trace element of Campanian - Maastrichtian Shale sediments exposed at Ahoko and Ojodu Town.

Trace element(ppm)	Patti Formation					Enugu Formation							
	AH1	AH3A	AH4	AH5C	AH8B	OJ1	OJ2	OJ3	OJ4	OJ5	Average		PAAS
											Patti	Enugu	
Sc	23	13	16	12	20	7	5	4	5	4	16.8	5	16.2
V	115	164	168	88	78	59	41	45	69	66	122.6	56	150
Cr	153	143	122	82	97	45	28	28	45	42	119.4	37.6	
Co	41	16	12	18	5	2	2	1	6	7	18.4	3.6	23
Ni	65	37	22	19	16	2	2	3	8	2	31.8	3.4	55
Cu	17	4	1	1	6	1	1	1	1	1	5.8	1	50
Zn	210	65	43	64	28	14	10	7	18	9	82	11.6	85
As	29	34	23	18	15	11	10	9	12	11	23.8	10.6	
Rb	40	52	48	47	42	18	16	18	20	22	45.8	18.8	160
Sr	248	240	150	61	58	34	35	39	34	69	151.4	42.2	200
Y	78	53	40	68	75	28	16	17	26	34	62.8	24.2	
Zr	767	652	534	1471	1926	1127	608	565	1006	1161	1070	893	210
Nb	66	64	49	53	58	19	12	13	19	22	58	17	1.9
Ba	578	568	419	346	399	209	216	223	193	273	462	222.8	650
Pb	1.5	1.5	3	6	15	1.5	1.5	1.5	1.5	8	5.4	2.8	20
Th	45	30	26	31	43	18	12	9	15	12	35	13.2	14.6
U	15	8	11	12	12	1.5	1.5	1.5	1.5	4	11.6	2	3.1

Table 4
Range of shale element ratios in this study area compared to ratios for similar fractions derived from post-Archean Australia shale.

Trace element Ratio	Enugu Average	Patti Average	PAAS
V/Ni	21.39	4.67	2.7
V/Cr	1.50	1.03	–
Ni/Co	1.32	2.00	2.3
V/(V + Ni)	0.95	0.80	0.7
Cr/Ni	14.49	4.43	–
Th/Co	5.64	3.09	0.6
Th/Sc	2.64	2.13	0.9

dominates shallow marine environment where there are low salt contents, this confirms a shallow marine environment for the shale of Enugu and Patti Formation. The absence of calcareous benthonic species and the presence of planktonic foraminifera are characteristics of anoxic bottom conditions, but agglutinated benthic foraminifera are resistant to dysoxic to anoxic conditions (Kaiho, 1994; Koutsoukos et al., 1990). Most of the foraminifera recovered from Enugu and Patti Formations are agglutinated, so dyoxic-anoxic environment is confirmed for their deposition.

Vanadium (V) and Nickel (Ni) presence in sediments are important index of the redox conditions that occur during sediments deposition. Anoxic marine environment is known to be rich in V when compares with Ni, this is because organic rich shale deposited under reducing conditions are always rich in V (Galarraga et al., 2008). Consequently, the plot of Vanadium against Nickel after Galarraga et al. (2008) (Fig. 12) shows that the sediments of Enugu and Patti formation is suggested to be marine anoxic environment. V/Ni ratios are also important paleoenvironmental indicator, ratio greater than 3 is attributed to reducing environment, while ratios ranging between 1 and 3 is attributed to suboxic – oxic conditions (Barwise, 1990; Ernst, 1970). The sediments of the Enugu and Patti formations both has V/Ni ratio greater than 3 suggesting a reducing (anoxic) environment for these two formations. Also, low value of Vanadium present in both formations support the continental detrital contributions in the deposition of the sediments (Ojo et al., 2016).

The V/Cr ratio also serves as a useful indicator for ancient redox conditions. According to (Dill et al., 1988), when the V/Cr ratio exceeds 2.0, it suggests the presence of anoxic conditions, while values below 2.0 indicate higher oxygen levels. The studied shales have V/Cr ratio less than 2 (Table 4) also suggests anoxic condition.

5.3. Paleotectonic

Studying paleotectonic environments through geochemical methods holds significant importance. Previous research, exemplified by (Bhatia and Crook, 1986; Roser and Korsch, 1986, 1988), has demonstrated that the chemical makeup of siliciclastic sedimentary rocks is heavily influenced by the tectonic conditions of their origins and deposition sites. Consequently, rocks sourced from distinct tectonic settings exhibit

Table 5
Relative Abundance of the mineral phases (%) as determined by XRD Analysis.

MINERALS	Patti Formation					Enugu Formation				
	AH1	AH3A	AH4	AH5C	AH8B	OJ1	OJ2	OJ3	OJ4	OJ5
Quartz	53	44	47	53	53	70	77	76	67	73
Kaolinite	16	34	30	19	22	12	11	10	22	10
Goethite	7				–					
Jarosite	6	6	6	8				2	2	11
Smectite	6									
Mica	5	12	13	16	17	7	9	4	8	5
Anatase	3	2	2	2	1					
K-feldspar/rutile	2	2	2	2	5	7	3	8	1	1
Siderite	2				2	2				
Calcite						2				

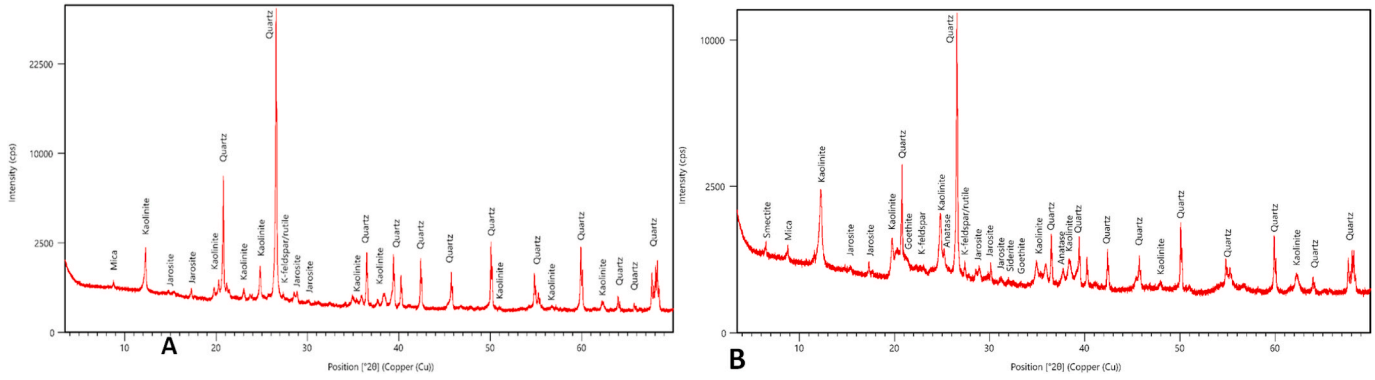


Fig. 8. X-ray diffraction diagrams for the Shale sediment of (A) Enugu Formation (B) Patti Formation.

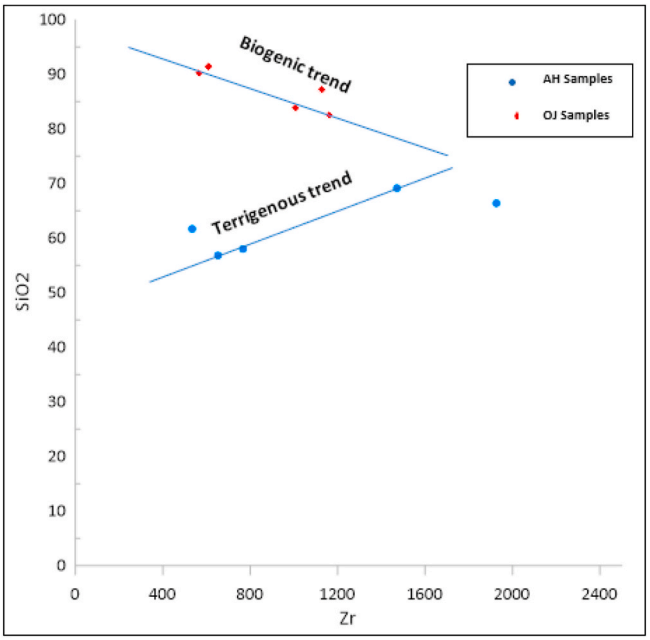


Fig. 9. Plot of SiO_2 versus Zr of shales from Ahoko and Ojodu (modified after Wright et al., 2010).

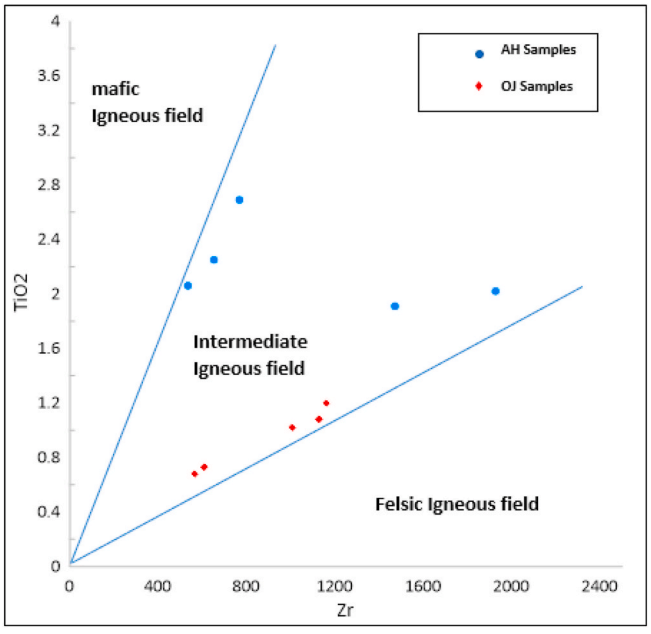


Fig. 10. Plot of TiO_2 versus Zircon of shales from Ahoko and Ojodu (modified after Ofili et al., 2022).

unique geochemical characteristics specific to their respective terrains. Major oxides, particularly those abundant in immobile forms like SiO_2 and TiO_2, along with reduced levels of mobile phases such as Al_2O_3, K_2O, CaO, and Na_2O, as well as their respective ratios (e.g., Al_2O_3/SiO_2, K_2O/Na_2O, $Al_2O_3/(CaO + Na_2O)$), serve as valuable geochemical indicators for deducing paleotectonic environments in clastic sedimentary (Bhatia, 1983; Crook, 1974; S. M. McLennan et al., 1990; Roser and Korsch, 1986). In this study we used geochemical plot of TiO_2 Vs Fe_2O_3+MgO, log (K_2O/Na_2O) vs SiO_2 and Al_2O_3/SiO_2 Vs Fe_2O_3+MgO (Figs. 13–15) modified after (Bhatia, 1983; Roser and Korsch, 1986) to deduce passive margin tectonic setting for the shale of Enugu and Patti Formations.

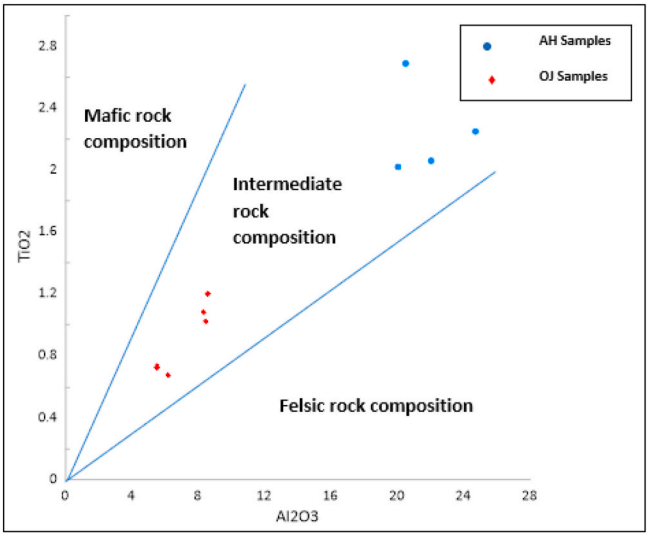


Fig. 11. Plot of TiO_2 versus Al_2O_3 of shales from Ahoko and Ojodu (modified after Ofili et al., 2022).

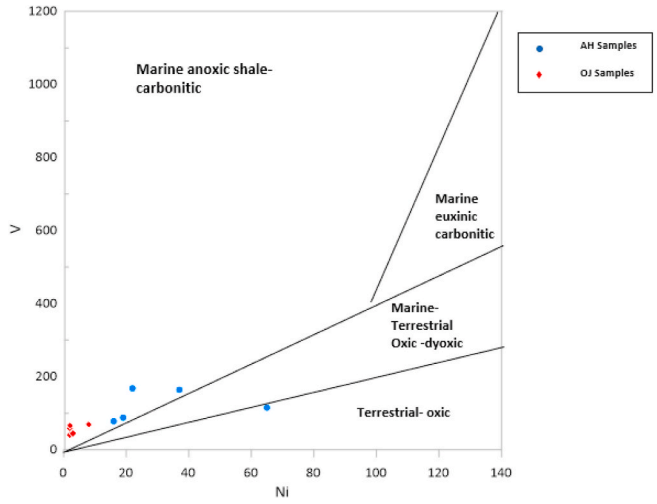


Fig. 12. Cross plot of Vanadium versus Nickel of shales from Ahoko and Ojodu (modified after Galarraga et al., 2008).

6. Conclusion

The results of the geological studies and laboratory analysis show that exposed sedimentary sections of Enugu formation in the Anambra basin and Patti formation in southern Bida basin consist similar lithologies from base to top. The Benthic foraminifera recovered from the samples are similar in these two basins. Presence of few monotonous species of arenaceous species are more in Anambra basin. Agglutinated benthic foraminifera such as *Haplophragmoides, Ammobaculites* and *Ammotium* suggests a brackish water in a shallow marine environmental setting for the shale of the studied basin. Also, organic content is low in both of the selected basins because of the low diversity and population of the recovered foraminifera species.

The study between the shale facies of the Enugu shale and Patti shale in southern Bida basin also displayed similar chemical and mineralogical composition with varying characteristics in the basins under study. Consequently, the results suggest sediments were deposited in shallow Marine environment in the two basins. However, there was influx of sediments from the continent in the Bida basin, and Enugu shale shows a negative correlation which can be as a result of biogenic substance from

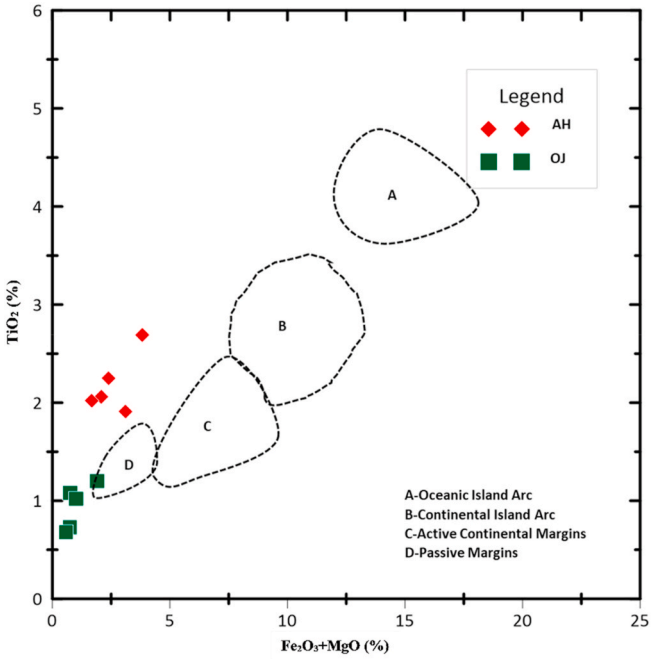


Fig. 13. Bivariate discriminate paleotectonic plots of TiO_2 Vs Fe_2O_3+MgO (modified after Bhatia, 1983).

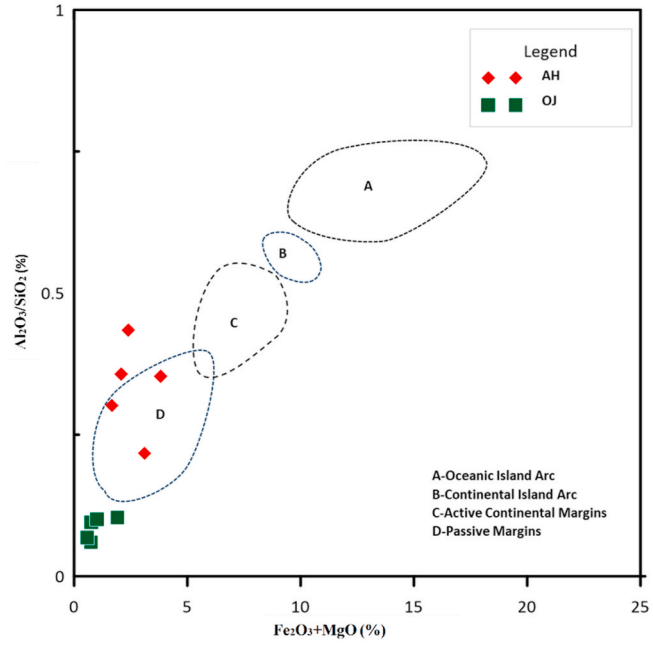


Fig. 14. Bivariate discriminate paleotectonic plots of Al_2O_3/SiO_2 Vs Fe_2O_3+MgO after (modified after Bhatia, 1983).

the Atlantic Ocean, but positive correlation from the Patti formation shows a continental influence from the Tethys Sea. Geochemical values such as V/Ni, V/Cr ratios derived suggests that the shales studied were deposited in an anoxic environment.

The geological, and paleontological studies, and inorganic geochemistry analysis have proved similar geological processes and depositional environments for the Campanian-Maastrichtian shale sediments in the Enugu and Patti Formations.

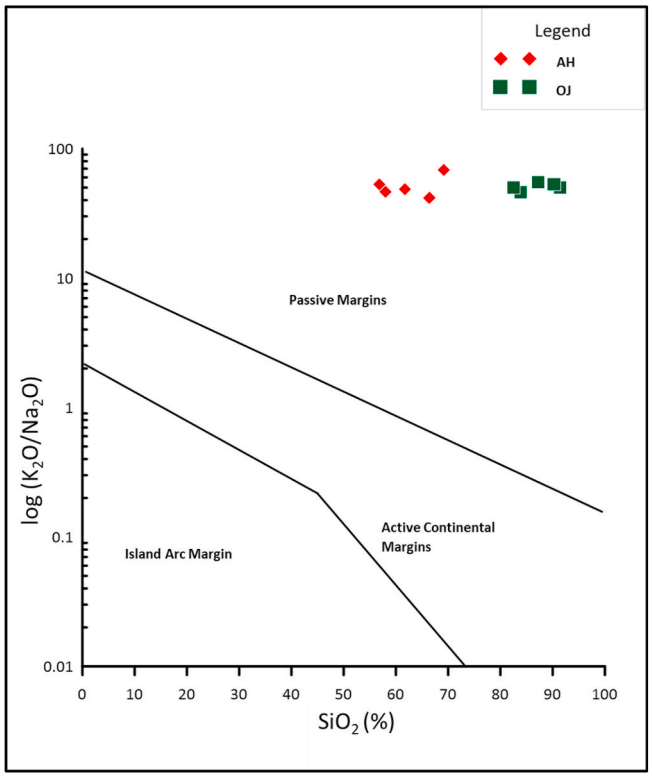


Fig. 15. K_2O/Na_2O vs SiO_2 (modified after Roser and Korsch (1986).

CRediT authorship contribution statement

Mariam Bolaji: Methodology, Investigation, Formal analysis, Funding acquisition, Project administration, Resources, Writing – original draft, Writing – review & editing. **Olabisi Adeleye Adekeye:** Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

References

Adeleye, D.R., 1974. Sedimentology of the fluvial Bida sandstones (Cretaceous), Nigeria. Sediment. Geol. 12, 1–24.

Adeleye, D.R., 1989. The geology of the mid- Niger basin. In: Kogbe, C.A. (Ed.), Geology of Nigeria, second ed. Elizabethan Publishing Co.

Adeoye, J.A., Akande, S.O., Adekeye, O.A., Abikoye, V.T., 2020. Geochemistry and paleoecology of shales from the Cenomanian-Turonian Afowo formation Dahomey basin, Nigeria: Implication for provenance and paleoenvironments. J. Afr. Earth Sci. 169, 103887 https://doi.org/10.1016/j.jafrearsci.2020.103887.

Agyingi, C.M., 1991. Geology of Upper Cretaceous Rocks in the Eastern Bida Basin, Central Nigeria. University of Ibadan, Ibadan, Nigeria. Ph.D. dissertation.

Akande, S.O., Erdtmann, B.D., 1998. Burial metamorphism (thermal maturation) in Cretaceous sediments of the southern Benue trough and Anambra basin. Nigeria: AAPG (Am. Assoc. Pet. Geol.) Bull. 82 (6), 1191–1206.

Akande, S.O., Ojo, O.J., Erdtmann, B.D., Hetenyi, M., 2005. Paleoenvironments, organic Petrology and rock eval studies on source rock facies of the Campanian to Maastrichtian Patti Formation, southern Bida Basin, Nigeria. J. Afr. Earth Sci. 41, 394–406.

Armstrong-Altrin, J.S., Lee, Y.I., Verma, S.P., Ramasamy, S., 2004. Geochemistry of sandstones from the upper Miocene Kudankulam Formation, southern India: implications for provenance, weathering, and tectonic setting. J. Sediment. Res. 74 (2), 285–297.

Barwise, A.J.G., 1990. Role of Nickel and Vanadium in petroleum classification. Energy & Fuels 4, 647–652.

Bhatia, M.R., 1983. Plate tectonics and geochemical composition of sandstones. J. Geol. 91, 611–627.

Bhatia, M.R., Crook, K.A.W., 1986. Trace element characteristics of graywackes and tectonic setting discrimination of sedimentary basins. Contrib. Mineral. Petrol. 92, 181–193.

Bolli, H.M., Saunders, S.B., 1985. Oligocene to Holocene low latitude planktic foraminiferal. In: M Bolli, H., B Sanders, J., Perch- nielsen, K. (Eds.), Plankton Stratigraphy. Cambridge University Press, pp. 155–262.

Bolli, H.M., 1957. The general Globigerina and Globorotalia in the Paleocene-lower Eocene Lizard Spring formation of Trinidad. United States. Bull. U.S. Natl. Mus. 215, 61–81.

Bolli, H.M., 1966. Zonation of Cretaceous to Pliocene marine sediments based on Planktonic foraminifera. Boletino informative Association Venezuela Geologia. Mineria Petrole 9, 3–32.

Condie, K.C., Liu, J., Quian, X., 1992. The origin of khondalites: geochemical evidence from the Archean to Early Proterozoic granulitic belt in the North China Craton. Precambrian Res. 59 (3–4), 207–223.

Crook, K.A.W., 1974. Lithogenesis and geotectonics: the significance of compositional variation in flysch arenites (graywackes). In: Dott Jr., , H, R., Shaver, R.H. (Eds.), Modern and Ancient Geosynclinal Sedimentation, vol. 19. SEPM Special Publication, pp. 304–310.

Cullers, R.L., 1995. The controls on the major and trace element evolution of shales, siltstones and sandstones of Ordovician to Tertiary age in the Wet Mountain region, Colorado, U.S.A. Chem. Geol. 123 (1–4), 107–131.

Dill, H., Teschner, M., Wehner, H., 1988. Petrography, inorganic and organic geochemistry of Lower Permian carbonaceous fan sequences ("Brandschiefer Series") — Federal Republic of Germany: Constraints to their paleogeography and assessment of their source rock potential. Chem. Geol. 67 (3–4), 307–325. https://doi.org/10.1016/0009-2541(88)90136-2.

Ernst, T.W., 1970. Geochemical Facies Analysis, first ed. Elsevier.

Galarraga, F., Reategui, K., Martïnez, A., Martínez, M., Llamas, J.F., Márquez, G., 2008. V/Ni ratio as a parameter in paleoenvironmental characterization of non-mature medium-crude oils from several Latin American basins. J. Petrol. Sci. Eng. 61, 9–14.

Gu, X.X., Liu, J.M., Zheng, M.H., Tang, J.X., Qi, L., 2002. Provenance and tectonic setting of the Proterozoic turbidites in Hunan, South China: geochemical evidence. J. Sediment. Res. 72 (3), 393–407.

Hamza, H., Obaje, N.G., Moumouni, A., 2011. Benthonic Foraminiferal Assemblages from the Bornu Basin, Northeastern Nigeria. J. Min. Geol. 48 (2), 91–115.

Hayashi, K., Fujisawa, H., Holland, H., Ohmoto, H., 1997. Geochemistry of ~1.9 Ga sedimentary rocks from northeastern Labrador. Canada: Geochimica et Cosmochimica 61 (19), 4115–4137.

Hossain, H.M.Z., Roser, B.P., Kimura, J.I., 2010. Petrography and whole-rock geochemistry of the Tertiary Sylhet succession, northeastern Bengal Basin, Bangladesh: provenance and source area weathering. Sed. Geol. 228, 171–183.

Jones, H.A., 1955. The Oolitic Ironstone of Agbaja Plateau, Kabba Province. Record of Geological Survey Nigeria (printed in 1958).

Jorge, R.C.G.S., Fernandes, P., Rodrigues, B., Pereira, Z., Oliveira, J.T., 2013. Geochemistry and provenance of the Carboniferous Baixo Alentejo Flysch Group, South Portuguese Zone. Sed. Geol. 133–148.

Kaiho, K., 1994. Benthic foraminiferal dissolved - oxygen index and dissolved -oxygen levels in modern ocean. Geology 22, 719–722.

Kennedy, W.Q., 1965. The influence of basement structure on the evolution of the coastal (Mesozoic and Tertiary) basins. In: Recent Basins Around Africa. Proceedings of the Institute of Petroleum Geologists Society, London, pp. 35–47.

King, L.C., 1950. Outline and distribution of Gondwanaland. Geol. Mag. 87, 353–359.

Koutsoukos, E.A.M., Leary, P.N., Hart, M.B., 1990. Latest Cenomanian to earliest Turinian low -oxygen tolerant benthonic foraminifera: case-study from Sergie Basin (N. E. Brazil) and western Anglo-Paris Basin (southern England). Paleogeography, Paleoclimatology, Paleoecology 77, 145–177.

Leventhal, J.S., 1998. Metal-rich black shales: formation, economic geology and environmental considerations. In: Schieber, J., Zimmerle, W., Sethi, P. (Eds.), Shales and Mudstones II. E. Schweizerbart'sche Verlagsbuchhandlung, Stuttgart, pp. 255–282.

McLennan, S.M., Hemming, S., McDaniel, D.K., Hanson, G.N., 1993. Geochemical approaches to sedimentation, provenance, and tectonics. Geol. Soc. Am. Spec. Pap. 284, 21–40.

McLennan, S.M., Taylor, S.R., McCulloch, M.T., Maynard, J.B., 1990. Geochemical and Nd–Sr isotopic composition of deep-sea turbidites: crustal evolution and plate tectonic associations. Geochim. Cosmochim. 54, 2015–2050.

Murray, J.W., 1991. Ecology and Paleoecology of Benthic Foraminifera. Longman Scientific & Technical, Harlow.

Nton, M.E., Bankole, S.A., 2013. Sedimentological characteristics, provenance and hydrocarbon potential of Post Santonian sediments in Anambra Basin, Southeastern Nigeria. RMZ – M&G 60, 47–66.

Nwajide, C.S., 1990. Cretaceous Sedimentation and Paleogeography of the Central Benue Though. In: Ofoegbu, C.O. (Ed.), The Benue. Tough Structure and Evolution International Monograph Series. Braunschweig: Berlin, Germany.

Obaje, N.G., 2009. In: Bhattacharji, B.S., Neugebauer, B.H.J., Reitner, G.J., Stuwe, G.K. (Eds.), Geology and Mineral Resources of Nigeria. Springer, Dordrecht Heidelberg London New York. https://doi.org/10.1007/978-3-540-92685-6.

Obaje, N.G., Moumouni, A., Goki, N.G., Chaanda, M.S., 2011. Stratigraphy, paleogeography and hydrocarbon resource potentials of the Bida Basin in North-Central Nigeria. J. Min. Geol. 47 (2), 97–114.

Obi, C.G., 2000. Depositional Model for the Campanian-Maastrichtian Succession, Anambra Basin, Southeastern Nigeria. University of Nigeria, Nsukka, Nigeria. Ph.D. Thesis.

Ofili, S., Soesoo, A., Panova, E.G., Hints, R., Hade, S., Ainsaar, L., 2022. Geochemical Reconstruction of the Provenance, Tectonic Setting and Paleoweathering of Lower Paleozoic Black Shales from Northern Europe. Minerals 12 (5), 602. https://doi.org/10.3390/min12050602.

Ogbahon, O.A., Olujinmi, O.B., 2019. Geochemistry of Maastrichtian Clastic Sedimentary Rocks from Western Flank of Anambra Basin, Nigeria: Implications for Provenance, Tectonic Setting, Paleoclimate and Depositional Paleoenvironment. Int. J. Geosci. 10 (1), 91–118. https://doi.org/10.4236/ijg.2019.101007.

Ojo, O.J., Akande, S.O., 2003. Facies relationships and depositional environments of the Upper Cretaceous Lokoja formation in the Bida Basin, Nigeria. J. Min. Geol. 39 (1) https://doi.org/10.4314/jmg.v39i1.18789.

Ojo, O.J., Babatunde, F.R., Adepoju, S.A., Akande, S.O., Momoh, A., 2016. Provenance and Weathering History of the Maastrichtian Shale Member of the Patti Formation, Southern Bida Basin, Nigeria. J. Geol. Soc. India 88, 471–480.

Ojo, S.B., Ajakaiye, D.E., 1989. Preliminary interpretation of gravity measurement in the middle Niger Basin area, Nigeria. In: Kogbe, C.A. (Ed.), Geology of Nigeria, second ed. Elizabeth Publishing Co, pp. 347–358.

Olabode, S.O., 2016. Soft Sediment Deformation Structures in the Maastrichtian Patti Formation, Southern Bida Basin Nigeria: Implications for the Assessment of Endogenic Triggers in the Maastrichtian Sedimentary Record. Open J. Geol. 6 (6), 410–438. https://doi.org/10.4236/ojg.2016.66036.

Pettijohn, F.J., 1975. Sedimentary Rocks, third ed. Harper & Row.

Reyment, R.A., 1965. Aspects of Geology of Nigeria. Ibadan University press.

Roser, B.P., Korsch, R.J., 1986. Determination of tectonic setting of sandstone-mudstone suites using SiO2 content and K2O/Na2O ratio. J. Geol. 94, 635–650.

Roser, B.P., Korsch, R.J., 1988. Provenance signatures of sandstone-mudstone suites determined using discrimination function analysis of major-element data. Chem. Geol. 67, 119–139.

Singh, P., 2009. Major, trace and REE geochemistry of the Ganga River sediments: influence of provenance and sedimentary processes. Chem. Geol. 266, 242–255.

Taylor, S.R., Mclennan, S., 1985. The Continental Crust: its Composition and Evolution. Blackwell, Oxford.

Tijani, M.N., Nton, M.E., Kitagawa, R., 2010. Textural and geochemical characteristics of the Ajali Sandstone, Anambra basin, SE Nigeria: Implication for Its provenance. C. R. Geoscience 342, 136–150.

Tribovillard, N., Algeo, T.J., Lyons, T., Riboulleau, A., 2006. Trace metals as paleoredox and paleoproductivity proxies: An update. Chem. Geol. 232 (1–2), 12–32. https://doi.org/10.1016/j.chemgeo.2006.02.012.

Uzoegbu, M.U., Ikwuagwu, C.S., 2016. Characterization of Organic Matter and Hydrocarbon Potential of Shale from Uturu, Isigwuato, Se Nigeria. International Journal of Scientific and Research Publications 6 (9), 754–759.

Whiteman, A.J., 1982. Nigeria, its petroleum geology, resources and potential. Graham and Trotman, London, U.K 1 (176), 2.

Wright, A.M., Spain, D., Ratcliffe, K.T., 2010. Application of Inorganic Whole Rock 2 Geochemistry to Shale Resource Plays, vol. 3. Society of Petroleum Engineers, pp. 1–18.