

TRACE ELEMENTS GEOCHEMISTRY OF FINE-GRAINED SEDIMENTS FROM THE MAMU FORMATION (ANAMBRA BASIN, NIGERIA): IMPLICATIONS FOR PALEOCLIMATE, SALINITY AND REDOX CONDITIONS

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ABSTRACT

Seven samples of silty muds and a coal sample were collected from two outcropping locations of the Mamu Formation of the Anambra Basin in order to reconstruct paleoclimate, paleosalinity and paleoredox depositional conditions of the sediments using discriminating geochemical indices. The samples were subjected to Energy Dispersed X-ray Fluorescence (EDXRF) analysis in order to reveal major trace elemental concentrations. With respect to Post Archean Australian Shales (PAAS), enriched trace elements in the fine grained sediments includes V, Cr, Cu, Ga, Zr and Zn, while depleted elements are Sr, Ba, Co, Ni and Rb. Sr/Cu ratios ranges from 0.393 – 3.75, with an average of 1.105, while Rb/Sr ratios ranges from 0.023 – 0.69 with a mean of 0.251. These values suggests a warm and humid climate with very minor influence of arid conditions. The Sr/Ba ratios ranged between 0.28 – 3.03, averaging 27.60, while Ba/Ga ratios ranges from 1.37 – 82.27 with a mean of 27.60, indicating a predominantly marine conditions interspersed with minor episodes of brackish and freshwater. The V/Cr ratios of the samples ranges between 0.17 – 3.39 with an average of 1.72, while ratios Ni/Co ranged from 0.252 – 4.63, with a mean of 3.92, suggestin a more oxic depositional setting with minor dysoxic and anoxic events

Keywords: Mamu Formation, Paleodepositional conditions, Salinity, Redox

Introduction

The geologic history of a sedimentary basin is vital in the search and exploration of mineral deposits. Elemental concentration in clastic and non clastic sediments has been used to constrain various geological parameters. Major elemental oxides like CaO, Na₂O, MnO, K₂O, Fe₂O and MgO are very unstable and tend to lose their percentage concentration in sediments during transportation, deposition and diagenesis. Hence, their geochemical ratios and proxies are mostly used to determine weathering, transportation history and lithological provenance in different tectonic setting (McLennan et al, 1993, Cullers, 1994; Armstrong altrin J. 2005). Due to their resistant and stable nature under subaerial conditions, trace elements are used to constrain other geologically important aspects of basinal studies, including paleoclimate, primary productivity, paleosalinity, and paleoredox conditions (Wang et al, 2024). The application of their geochemical finger prints is very effective in mostly fine grained sediments including shales, mudstones, siltstones and other non clastics like coals and lignites.

Documented geochemical studies on the outcropping sediments of the Mamu Formation includes Okiotor and Igodaro (2020), Ogala (2020), Odomar and Idakwor (2020), Akinyemi *et al*, (2022) and others. These investigations used both major and trace elemental concentration to evaluate lithological provenance, paleoclimatic, paleoredox, paleosalinity, paleoproductivity and depositional tectonics on the sediments of the Mamu Formation outcropping mostly at the western flank of the Anambra Basin

Trace elements geochemical evaluation of the Mastrichtian sediments of the Mamu Formation in thw eastern axis of the Anambra Basin is grossely inadequate , leading to deficits of very important aspects of basinal history useful for geological modelling and exploration purposes. In other to fill this existing gap this work is focused on the application of established trace element geochemical indices for the determination of paleoclimate, paleoredox and paleosalinity conditions of the fine grained muddy facies of the Mamu Formation outcrops located in the Enugu Axis of Anambra Basin. The study hopes to provide a broader concept and understanding on the paleo-depositional environments of the sediments using only trace elements which would compliment results of previous research on the sediments of the Mamu Formation.

Geologic and Stratigraphic setting

The origin of many sedimentary basins in Nigeria is tied to the separation of the South American and African plates 65mys ago, which began with the development of a tripple junction, off the Gulf of Guinea (Bankhelil, 1971). This event heralded the opening of the Benue Trough, forming the Abakaliki- Benue fold belt (Whiteman, 1982), which resulted from the failed arm of the “trippel” junction (aulacogen) following the development of “hotspot” (Burke and Dew, 1974, Olade, 1975).

The Santonian thermotectonic event led to the formation of the Abakliki high, laterally shifting the depocentre westward (Anambra Basin), and south eastward (Afikpo Sub-Basin) (Murat, 1972). The Abakaliki anticlinorium is formed by tightly folded Cretaceous sediments, intruded by numerous magmatic intrusions. The Anambra Basin is a roughly triangular shape NE -SW trending siliciclastic wedge, which covers a total area of approximately 40,000km², with sediments thickness increasing southwards to a maximum of roughly 600m ((Nwajide and Reijers, 1996) (Fig 1). Major sedimentation in the Anambra Basin began in the Campanian with a short marine transgression followed by regression. The basal lithostratigraphic unit of the basin’s sedimentary sequence is the Nkporo Group, which consists of Nkporo shale, Enugu shale and Owelli sandstone as its lateral equivalent (Reijers, 1998). The Nkporo shale is composed of dark gray, very fissile, but soft shales and sandstones with occasional thin inter beds of sandy shales,

sandstones and marls with sulphur coatings (Agagu et al,1985; Nwajide,2013).The formation is highly fossiliferous consisting of both marine and terrestrially derived faunas (Akhaegbobi and schmits,1988). Subsequent marine regression in the Maastrichtian resulted in the deposition of Mamu, Ajali and Nsukka Formation. The Maastrichtian Mamu Formation consists of alternation of fine to medium grained, well sorted sandstones, dirty grey shales and sandy shales, siltstones and bituminous coal seams (Nwajide, 2013). The occurrence of the coal suggests a deposition in a paludal, deltaic and possibly shallow marine setting (Adeniran, 1991). The Ajali Formation (false bedded sandstone) lies comfortably on Mamu Formation and consists of thick, friable, moderately sorted whitish medium to coarse, burrowed sandstones, typically white, with occasionally clays draped cross bedding (Umeji and Nwajide, 2007). Ajali sandstone is a continental fluvio-deltaic sequence characterized by a regressive phase of a short lived Maastrichtian transgression with sediments derived from westerly areas of Abakaliki anticlinorium and granitic basement unit of Adamawa – Oban - masiff, and The possessed both coarsening and fining upward sequence in places (Ladipo, et al,1992). The Nsukka Formation conformably overlies the Ajali sandstone.It occur extensively in the Anambra Basin, and marks the end of the Cretaceous sedimentation. Lithologically, it consists of coarse to medium grained sandstones, and passes upward into well bedded blue clays, fine grained sandstones and carbonaceous shales with thin beds of limestone (Nwajide, 2013) (Fig 2).

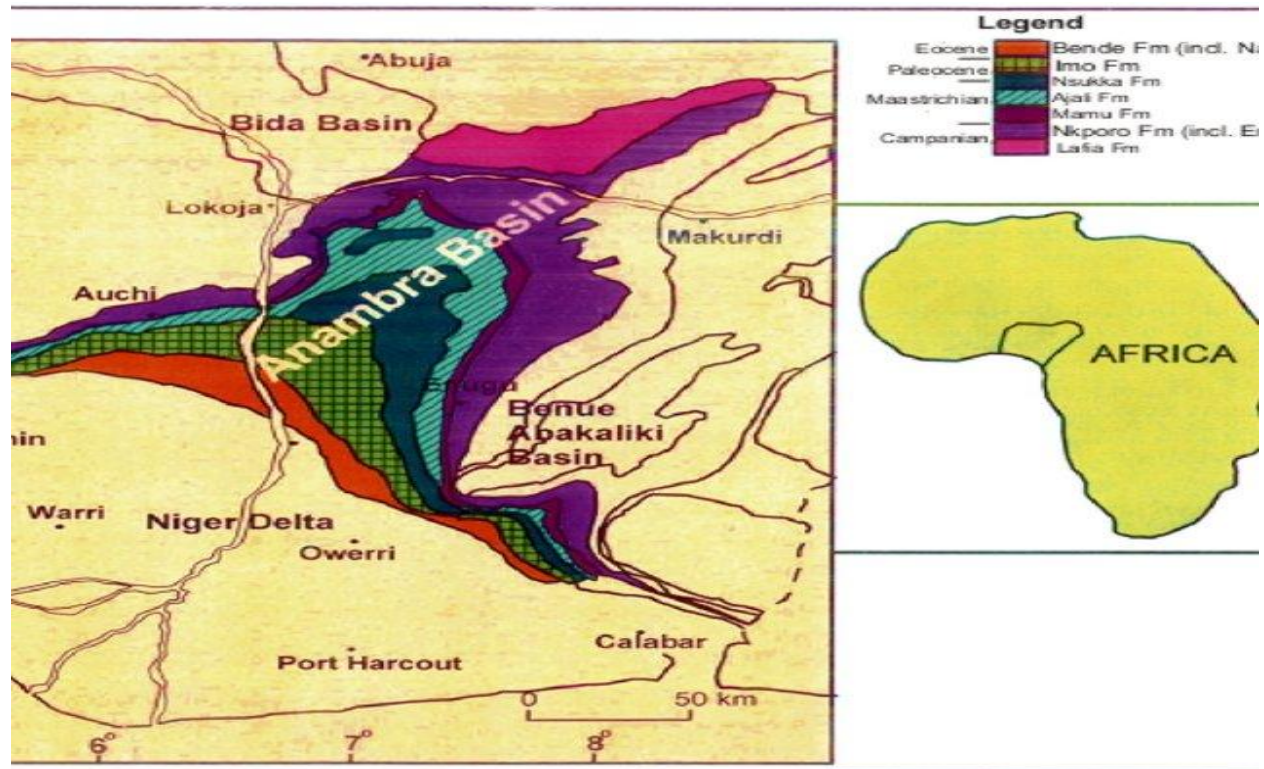


Fig 1; Geological map of the Anambra Basin (After Nwajide, 2003)

System / Series / Stage		Formation	Sedimentary environment	
Quaternary		Benin Formation	Deltaic/ Continental/ Shallow marine	
Neo- gene	Pliocene			
	Miocene	Ogwashi-Asaba Formation		
Paleogene	Oligocene	Ameki/Nanka/ Nsughe Sandstones		
	Eocene			
	Paleocene	Imo Formation Nsukka Formation		Shallow marine
Cretaceous	Maastrichtian	Ajali Formation Mamu Formation		Fluvio-deltaic/ Marginal marine
		Campanian		Nkporo/Oweli/ Enugu Shale
	Santonian	~~~~~~		
	Coniacian	Awgu Formation		Shallow marine
	Turonian	Eze-Aku Formation	Marine	
	Cenomanian	~~~~~~		
	Cenomanian	Odukpani Formation	Shallow marine	
	Albian	Asu River Group	Marine	
	Aptian Barremian Hauterivian	Unnamed units		
Precambrian		Basement complex		

Fig 2: Stratigraphy of the Anambra Basin (Modified from Nwajide, 1990)

Method of Study

The study areas are two outcrops of the Mamu Formation in Enugu. the first location is along the Ogbanikwute stream, off Enugu ninth mile expressway and designated as location one (L1), while the second location is along Evo road, off new market, Enugu (fig 3). The two locations are within the coordinates.

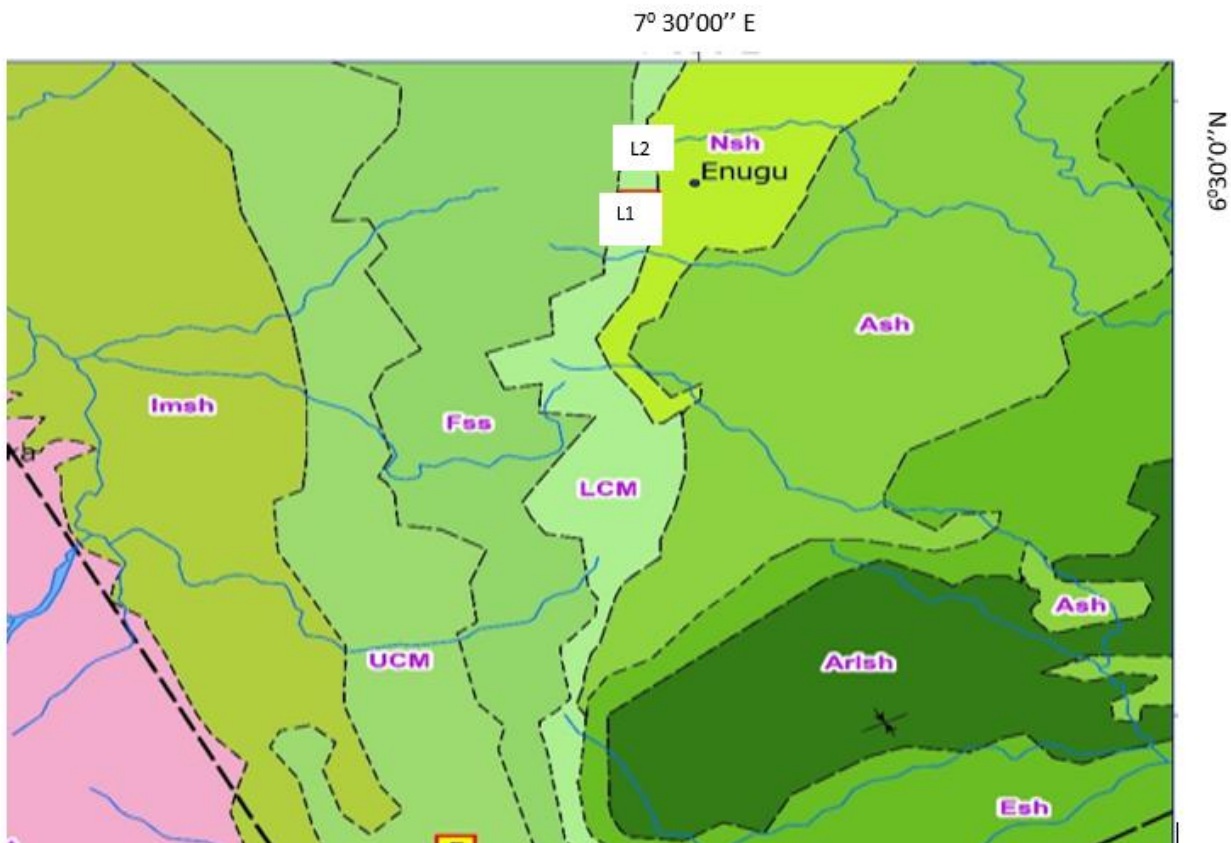
L-1 N = 6° 27' 52.9979"; E = 7° 29' 4.89984"

L2: N = 6° 28' 032"; E = 7° 27' 07.2"

The two locations, which are vertically and laterally extensive outcrops were visited during a two days field work. Location one (L1) consists of interbedded silty grey muds and very thin coal bed, while location two(L2)is made up of of thickly interbedded light grey silty muds, shaley muds and embedded red ironstones. A total of two samples of silty muds and a sample of coal were retrieved from the first location and labeled L1a, L1b and L1c. While five samples of fine grained, light to dark grey coloured silty muds were retrieved from two measured section of the second location and designated as L2a, L2b, L2c, L2d, and L2e, bringing the total samples to eight (8), which were bagged with cellophane and sent to the geochemistry laboratory of the geological survey agency (NGSA) for X-ray Fluorescence (XRF) analyses.

The samples were pulverized (grind to fine powder) using arget pulverizing machine (planetary micro mill pulverisette 7). The ground samples were made to pass 150 micro mesh sieves in other to ensure homogeneity of the samples. 5g of the pulverized sample was weighed into a beaker with 1g of binding aid (Starch soluble). The mixture was thoroughly mixed to ensure homogeneity, which was pressed under high pressure (6 “tone”) to produced pellets which were labeled and package for the analysis.

Energy Dispersive x-ray fluorescence (EDXRF) spectrometer model “Minipal 4” was used for the analysis. The pellets were carefully placed in the respective measuring positions on a sample changer of the machine. The following sets of condition were made as the machine was switched on (i):Elemental composition determination(i) Nature of the samples to analyzed as press powder (pellet).(iii)The current used is 14kv for major oxides, 20kv for the trace elements/rare earth metals(iv)Selected filters were “kapton” for major oxides, while Ag/Al-thin for the trace elements/rare earth metals.



KEY

Fss	False Bedded Sandstone
UCM	Upper Coal Measures
LCM	Lower Coal Measures
Nsh	Nkporo Shale
Ash	Awgu Shale
Esh	Ezeaku Shale
Arish	Asu River Group

Fig 3: Geological map of study area showing sampled locations

Results and Discussions

1. Geochemistry of the sediments

Table 1 shows the results of the X-Ray Florescence Analysis (XRF) of the fine grained sediments obtained in part per million. The selected trace elements in ppm includes Vanadium (V), Chromium (Cr), Copper (Cu), Strontium (Sr), zircon (Zr), Barium (Ba), Zinc (Zn), Cobalt (Co), Nicke (Ni), Gallium (Ga) and Rubidium (Rb).

It is important to note that sample L1c is a coal sample, while all others are silty muds as earlier stated.

The elemental ranges and their averages include: V (20 - 515, av=289.65); Cr (22. - 243, av = 196. 19); Cu (12.00 - 480. av = 263.91); Sr. (30.30 - 2060, av = 127.29); Ga (5.09 – 39.50, av.=33.28); Zr (103 -450, av= 224.25); Ba (10.00 - 940, av = 336.43); Zn (20.00 – 370, av = 120.5); Co (0.01- 1.5, av = 0.49); Ni (0.11-1.53.10, av = 0.57); and Rb (0.70 - 36.50, av = 26.81).

Elemental enrichment or depletion is a function of source area composition, degree of weathering, transportation history and severity of diagenetic alteration (Rosa and Korsch, 1986). With respect to Post Archean Australian shales (PAAS) (McLennan *et al.* 1993), the enriched trace elements in the fine grained sediments include V, Cr, Cu, Ga, Zr and Zn, while depleted trace elements are Sr, Ba, Co, Ni and Rb. The enriched trace elements may be attributed to their abundance in mafic and ultra-rocks which are very resistant to chemical destruction. . Secondly, their association with organic matter in a reducing environmental setting may also contribute significantly to their high concentration. Severity of chemical weathering, diagenesis may have led to the depletion of Sr, Ba, Co and Ni. Sr is very mobile element in an hydraulic medium, while Ni and Co are also have known association with paludal environments where coals and lignites are developed. .It has been noted that the concentration of trace elements in coals are highly variable. Higher rank coal coals like Anthracites and certain geological formations may have higher concentrations, while lower grade coals have much lower concentrations (Wang et al, 2024).

Table 1: XRF Result of Samples

Trace Elements (PPM)	L1a	L1b	L1c	L2a	L2b	L2c	L2d	L2e	AV	PAAS McLennan et al, 1993
V	20.00	82.00	35.20	401	431	412	421	515	289.65	120
Cr	22.70	458.40	36.80	118	135	243	326	233.19	196.63	110
Cu	12.00	252.80	12.55	480	334	330	380	310	263.91	50.0
Sr	45.00	79.00	30.30	189	162	206	183	124	127.29	200
Ga	5.50	121.10	6.60	34.02	39.50	22.00	5.09	32.50	33.28	19
Zr	450.00	254	20.00	571	124	152	120	103	224.25	210
Ba	110.4	167.00	10.00	670	311	182	301	940	336.43	650
Zn	173.00	108.00	20.00	100	97	56	370	40	120.5	85
Co	0.26	0.14	0.33	0.11	0.01	1.23	1.5	0.4	0.49	17
Ni	0.12	1.32	1.53	0.46	0.11	0.31	0.25	0.52	0.57	44
Rb	31.10	30.40	0.70	22.60	29.80	33.40	36.50	30.00	26.81	160

Paleoclimate

Trace elements are useful determinant of paleoclimatic conditions of siliciclastic sediments (Cao et al, 2012, Tao et al, 2017). It has been documented that Fe, Mn, V, Cr, Co and Ni are relatively enriched under humid climatic conditions, while Ca, K, Sr, Mg, and B are precipitated in arid conditions. Irrespective of the lithological characteristics, the most useful trace elemental proxies (ratios) for paleoclimatic discrimination are Sr/Cu and Rb/Sr ratios (Wang, 2021, Hamdani et al, 2021). The Sr/Cu ratio values increases in dry climates and decreases in warm and humid climates. Thus 1.3 – 5.0 indicates warm and humid climates, while > 5.0 suggests hot and dry climatic conditions (Lerman, 1978; Grant, 1984; Tao et al 2017). From the eight samples obtained from the two locations, almost all Sr/ Cu ratios values ranges from 0.313-3.75, with an average of 1.105, indicating a warm and humid climatic conditions (Table 2). Unlike the widely applicable Sr/Cu ratio, Rb/Sr must be used with caution, as its use in climatic discrimination is largely dependent on different climatic belts, which may be Monsoon, arid, semi humid or polar conditions. Within tropical regions, Rb/S ratios are very low in warm climatic, and high in cold climatic conditions respectively (Chen et al, 1999; Xu et al, 2010). The 0.251 average of samples suggests warm climatic conditions, implying deposition of the sediments (including the coal L1c) in warm and humid paleoclimate.

The Sr/Cu paleoclimatic discrimination diagram shows a migration of data points from the arid axis to a condensation at the warm humid segment. Which may suggest a short ranged initial paleoclimatic conditions to a rapid warm and humid climate during the deposition of the coal and the other fine grained silty muds (fig 4),

Table 2: Sr/Cu and Rb/Sr paleoclimatic Indicators of samples

Samples	Sr/Cu	Interpretation	Rb/Sr	Interpretation
L1a	3.75	Warm/ humid	0.69	Warm/humid
L1b	0.313	Warm/humid	0.38	Warm/humid
L1c	2.39	Warm/humid	0.023	Warm/humid
L2a	0.393	Warm/humid	0.12	Warm/humid
L2b	0.49	Warm/humid	0.18	Warm/humid
L2c	0.624	Warm/humid	0.162	Warm/humid
L2d	0.482	Warm/humid	0.294	Warm/humid
L2e	0.4	Warm/humid	0.164	Warm/humid
AV	1.105	Warm/humid	0.251	Warm/humid

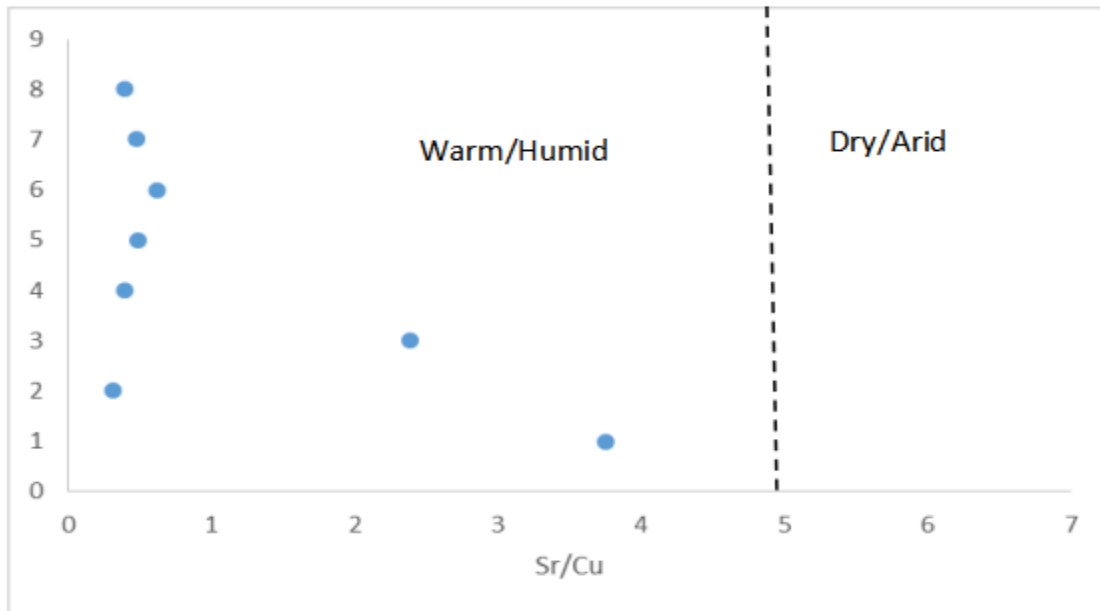


Fig 4: Sr/Cu paleoclimatic discrimination

Paleosalinity

Trace elemental ratios are also important proxies for determination of paleosalinity and depositional environments of siliciclastic sediments. Sr/ Ba and Ba/Ga are the most common ratios used to infer paleosalinity (Wei Wei and Algeo, 2019). Sr/ Ba ratios of <0.2 indicates fresh water; 0.2- 0.5 indicates brackish water and >0.5 indicates marine (Saline) water conditions. From table 3, three samples (L1a , L1b and L2a) records 0.41,0.47 and 0.28 respectively, suggesting brackish water conditions , while the other samples including the coal(L1c) reflects marine (Saline water) environment, with just a sample reflecting fresh water setting, implying a more marine environment with brackish and fresh water inputs. Secondly Ba/Ga values of <3 indicates fresh water; 3-6 indicates brackish water and >6 is a marine (saline) water environments. A total of five samples indicate saline water environment, while the coal and two other samples has values that suggests fresh water envoronment setting, indicating a predominatly marine inundation

interspersed with minor episodes of fresh water influence from fluvial sources . A binary plot of Sr vs Ba (Hamdani and Alam, 2021) indicates a scattering of the data points between a more marine setting with minor inputs of brackish and fresh water influence (Fig 5) This may reflects deposition of sediments in a water body which may have experienced a high rate of evaporation, resulting in a saline (marine) conditions with some dilution from fluvial systems and precipitation occasioned by climatic variation between Warm/ climate and shot- lived arid conditions.

Table 3: Sr/Ba and Ba/Ga paleosalinity interpretation of samples

SAMPLE	Sr/Ba	Salinity Interpretation	Ba/Ga	Salinity interpretation
L1a	0.41	Brackish	20.09	Marine
L1b	0.47	Brackish	1.37	Fresh
L1c	3.03	Marine	1.52	Fresh
L2a	0.28	Brackish	19.7	Fresh
L2b	0.52	Marine	7.87	Marine
L2c	1.13	Marine	82.27	Marine
L2d	0.61	Marine	59.13	Marine
L2e	0.13	Fresh	28.9	Marine
Average	0.81	Marine	27.60	Marine

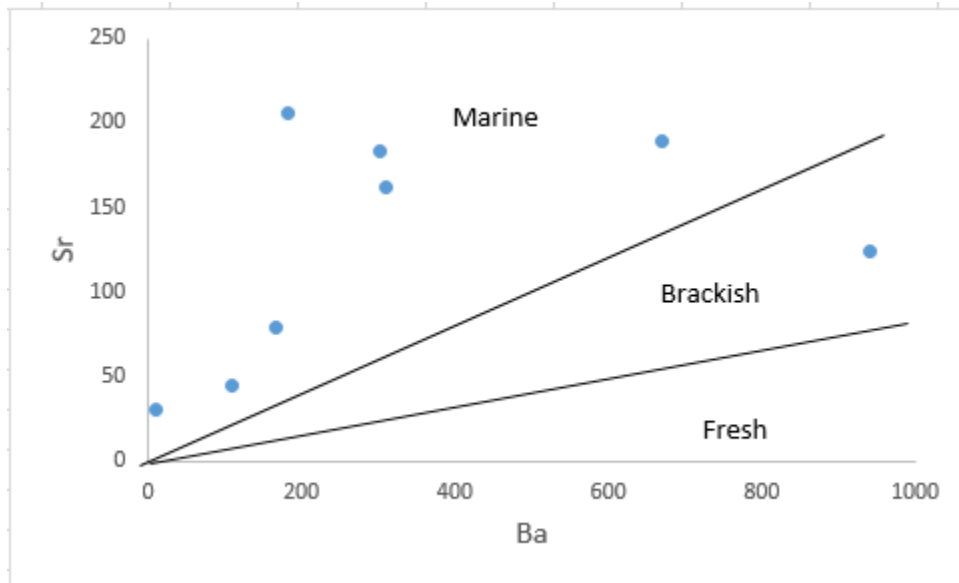


Fig 5: Binary plot of Sr and Ba salinity interpretation of samples (After Hamdani and Alam.2021)

Paleoredox Conditions

Redox conditions are generally classified into Oxidic (>2 ml O₂ L⁻¹), dysoxic (2 - 0 ml O₂ L⁻¹ mlO₂/LH₂O), anoxic (0 ml O₂ L⁻¹), and euxinic (0 ml O₂ L⁻¹, H₂S > 0) conditions (Tribollivard et al, 2006; Algeo and Liu, 2020). Paleoredox sensitive trace element includes Ni, V, Cr, Co, Cu and Zn. Jonnes and Mannings (1994) proposed V/Cr and Ni/Co ratios for the determination of paleoredox conditions of clastic sediments. They stated that V/Cr ratios of <2 indicates oxidic conditions, 2- 4.5 indicates dysoxic conditions and >4.25 indicates suboxic to anoxic conditions.

From table 4, Five (5) samples, including the coal (L1c) indicates oxic conditions, while three(3) showed a dysoxic conditions, suggesting a more oxic depositional medium with minor episodes of dysoxic environmental setting where dissolved oxygen is low. The ratio ranges of Ni/Co includes; <5 (Oxic); 5-7 (Dysoxic) and >7 (Suboxic/ anoxic conditions. Six (6) samples (including the coal L1c) showed a Ni/Co values that suggests oxic conditions, while two others indicates suboxic/Anoxic where oxygen level is slightly elevated . A cross plot of V/Cr and Ni/Co suggests predominantly oxic and less dysoxic/suboxic depositional medium of the fine grained sediments of the Mamu Formation (Fig 6). The interpretation suggests a partially enclosed shallow water body like lagoon, proximal part of an estuary or a lacustrine setting in a delta or coastal plain where water stratification exists.

Table 4:V/Cr and Ni/Co paleoredox interpretation of samples (After Jonnes and Mannings,1994)

SAMPLE	V/Cr	Interpretation	Ni/Co	Interpretation
L1a	0.88	Oxic	0.46	Oxic
L1b	0.17	Oxic	9.42	Suboxic/Anoxic
L1c	0.96	Oxic	4.63	Oxic
L2a	3.39	Dysoxic	4.18	Oxic
L2b	3.19	Dysoxic	11	Suboxic/ Anoxic
L2c	1.69	Oxic	0.252	Oxic
L2d	1.29	Oxic	0.16	Oxic
L2e	2.20	Dysoxic	1.3	Oxic
AV	1.72	Oxic	3.92	Oxic

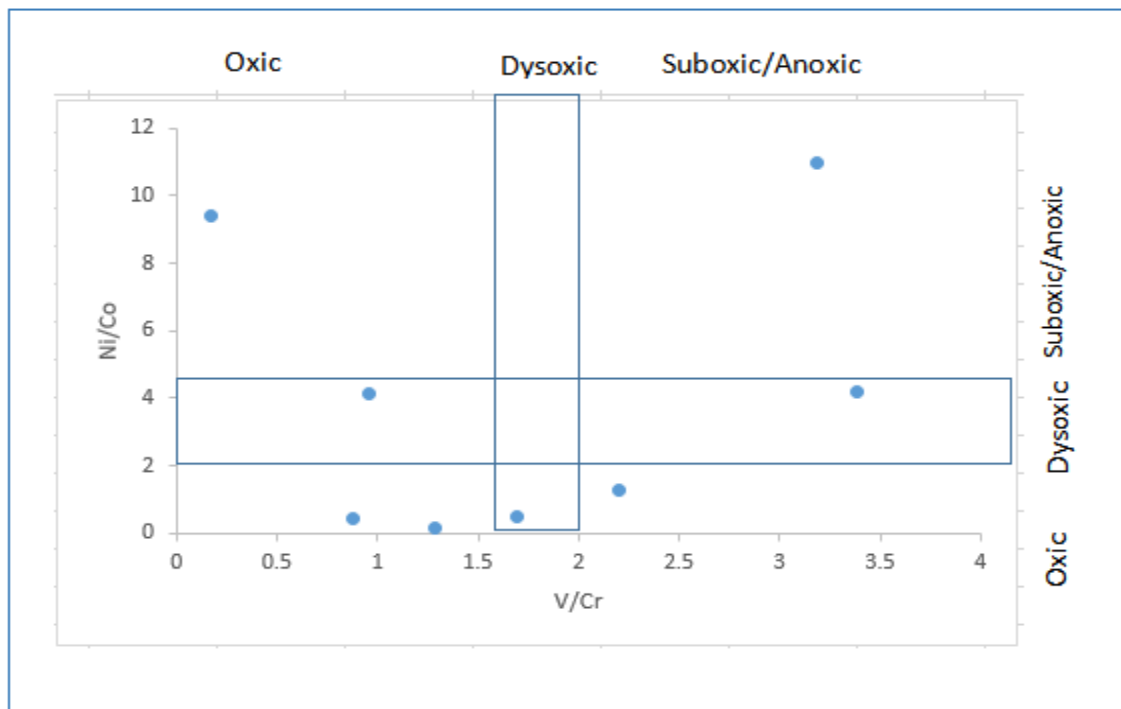


Fig 6: Binary plot of V/Cr and Ni/Co paleoredox condition of samples (After Jones and Mannings,1994)

Conclusion

One coal and seven other silty mud samples were collected from two outcrop of the Maastrichtian Mamu Formation at the Enugu axis of the Anambra Basin, in order to determine their paleodepositional conditions, including climate, salinity and redox status, using trace elemental geochemistry. The eight samples were subjected to Energy Disperse X- Ray Fluorescence Analysis (EDXRF) and results obtained in part per million(ppm).The averages of selected trace element concentration includes V(289.5),Cr (196.19), Cu(263.19) ,Sr (127.90), Ga (33.28), Zr (224.25), Ba (336.43), Zn (120.5), Co (0.49), Ni(0.57) and Rb(26.81). With respect to Post Archean Australian Shale (PAAS), V, Cr, Cu, Ga, Zr and Zn were enriched, while Sr, Ba, Co, Ni and Rb were depleted. It has been reported that enrichment or depletion of trace elements in fine grained sediments is a function of climatic types and intensity of chemical weathering, transportation and diagenetic finger printing. But their variation in coal may be due to coal ranks and paleoclimatic conditions. The Sr/Cu ratio varies between 0.393 – 375, with an average of 1.05 , while Rb/Sr ratios are between 0.023 – 0.69, with a mean value of 0.25. These values together with the discrimination plot reflects sediments that were deposited in warm and humid climate with minor input of aridity. The Sr/Ba proxy ranged between 0.13 - 3.03, with an average of 0.81, while Ba/ Ga ratios ranges between 1.37 – 82.27, averaging 27.60, suggesting a more marine depositional medium with little inputs from brackish and fresh water from fluvial systems. The V/Cr paleoredox proxy ranges from from 0.17 – 3.39 with an average of 1.7, while that of Ni/Co ratios ranges between 0.16 – 9.42, averaging 3.92. These values and the relevant plots points to a more oxic conditions where sea level variations may have introduced minor suboxic/ anoxic events. The depositional setting of the sediments reflects a shallow water body in a coastal or delta plain where a humid climate with high salinity and oxygen rich conditions persisted through geologic time. A more detailed geological methods integrating geophysical, sedimentological, biostratigraphical and major elements geochemistry of the Mamu Formation would increase very reliable datas for a better understanding of the geology that could utilized for economic purpose..

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