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Geochemical Investigation of Cretaceous Dukul Formation from Yola Sub-basin, Northern Benue Trough, NE Nigeria: Paleoenvironment, Paleoclimate and Tectonic Setting

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ABSTRACT
The Cretaceous sediments of Dukul Formation from Yola Sub-basin, Northern Benue Trough, north-eastern Nigeria were investigated based on geochemical parameters; mineralogical composition, major and trace elements geochemistry. This is with a view for assessment of the paleodepositional environment, paleoclimatic condition and tectonic setting during deposition of the sediments based on the application of X-ray diffraction (XRD), X-ray fluorescence (XRF) and Inductively Plasma Mass Spectrometry (ICPMS). The analysed samples contain minerals such as illite, glauconite, montmorillonite with predominance elements such as SiO2 followed by Fe2O3, CaO, Al2O3, and TiO2 whilst trace elements Sr, Ba, V, Ni, Co, Cr. The sediments were interpreted to have been deposited in a shallow marine (shelf) environment under suboxic/dysoxic marine depositional condition during the arid/sub-arid paleoclimatic conditions along with the passive continental margin setting. This paleoclimatic condition and tectonic setting were responsible for the deposition of most of the Cretaceous sediments within the West and Central African Rift System (WCARS) of which Benue Trough is inclusive.

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1.1 INTRODUCTION

The Northern or Upper Benue Trough (NBT) is an extension of the Nigerian Benue Trough. The trough is geologically sub-divided into the Gongola and Yola Sub-basins. The Yola Sub-basin consist of sequences of gravels, sandstones, shales and limestones. The sediments were deposited in continental to coastal and shallow marine depositional environments, ranging in age from Cretaceous to Cenozoic in association with Neogene volcanism (e.g. Nwajide, 2013; Carter *et al*., 1963; Abubakar, 2014; Sarki Yandoka *et al*., 2014). The study area (Fig. 1), the Cretaceous Dukul Formation was earlier recognized as "Limestone – Shale Series" sequences and was assigned to the Lower Turonian age (Carter *et al*., 1963; Grant, 1971; Guiraud, 1990, 1992; Sarki Yandoka, 2015). The formation consists of clays, shale, siltstones and thick limestone inter-bedded with shales (Nwajide, 2013; Sarki Yandoka, 2015). The sequences represent part of shallow-marine sedimentation in Yola Sub-basin and they are the lateral facies equivalent of the Kanawa Member (Zarborski *et al*., 1997; Abubakar, 2014; Sarki Yandoka, 2015; Sarki Yandoka *et al*., 2015a, 2015b, 2015c, 2016) in the Gongola Sub-basin.

The inorganic geochemical character of sedimentary rocks and basins are the products of many factors such as mineral composition, climate and tectonism (Suttner and Dutter, 1986). Detail geochemical assessment of the Dukul Formation sediments from Yola Sub-basin of the Northern Benue Trough based on inorganic geochemical characterisation in order to determine the paleoenvironment, paleoclimate and tectonic setting is lacking in the literature. More so, most of the published works are on lithofacies and organic facies characterisation. The present research, however, attempts to present the inorganic geochemical data (mineralogical, major and trace elemental composition) of sedimentary successions of Cretaceous Dukul Formation exposed in the Yola Sub-basin of the Northern Benue Trough with the objectives of determining the depositional environment, paleoclimatic conditions and tectonic setting during deposition of the sediments. The study is also important for paleodepositional environmental, tectonostratigraphic and sequence stratigraphic models of the entire Northern Benue Trough particularly in view of the current hydrocarbon exploration campaigns, resource assessment and risks evaluation of the region.

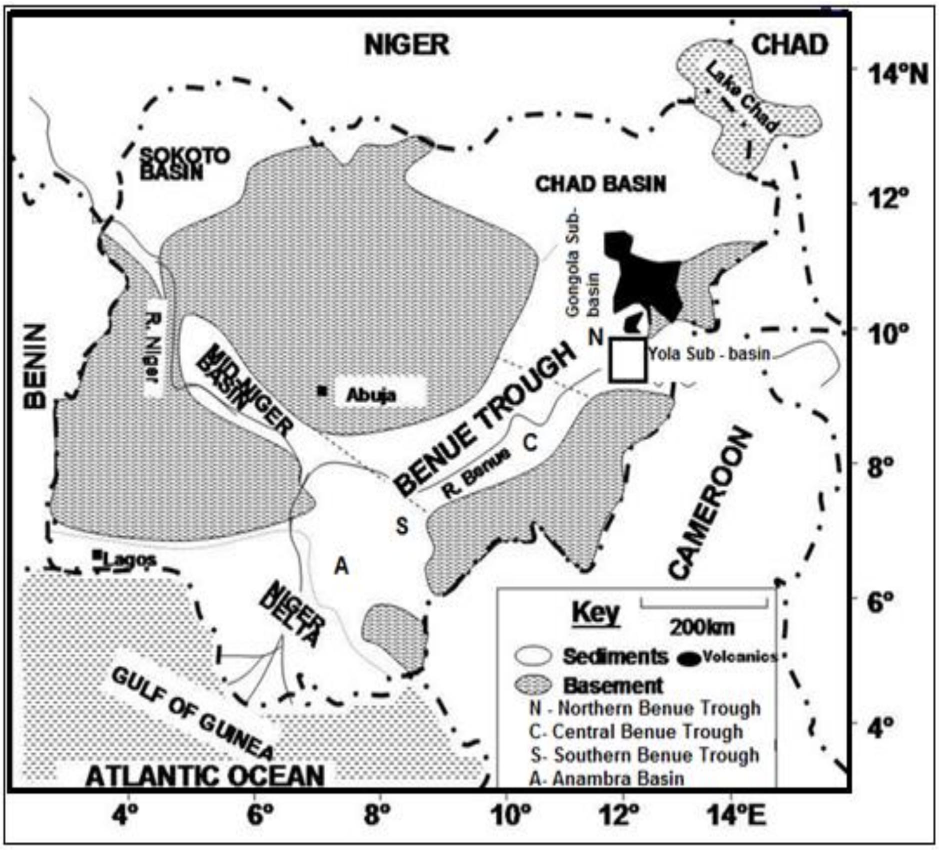

Figure 1: *Geological map of Nigeria showing the study area (Source:* Abubakar, 2014)

2.1 GEOLOGICAL SETTING

The Nigerian Benue Trough was formed during the Early Cretaceous rifting and strike-slip faulting or movement of the West and Central African Rift System (e.g. Carter *et al*., 1973; Abubakar, 2014; Benkhelil, 1982, 1989). Grant (1971), Benkhelil (1989) and among many others suggested rifting and strike-slip faulting as the basis for the evolution of the Benue Trough. The acceptable model is that of Grant, (1971) which suggested rifting model that formed at triple junction thereby, producing the third failed arm or aulocogen (RRF model) as supported by Zarborski *et al*., (1997).

The Benue Trough trends northeast-southwest for about 1000 km in length and about 150 km average in its width, bounded by Niger Delta Basin at the southern end of the trough and the Chad (Bornu) Basin in the northern end. The trough contains thick Cretaceous to Cenozoic sedimentary profile for up to 6000 m associated with volcanic of Neogene age. Based on geographical view, the Benue Trough is divided into Southern (Lower), Central (Middle) and Northern (Upper) Benue basins (Zaborski, *et al*., 1997; Nwajide, 2013; Sarki Yandoka *et al*., 2014; Sarki Yandoka, 2015) (Fig. 1).

The stratigraphy of Yola Sub-basin comprises the continental Bima Formation, consisting of cobbles, gravels, sandstones and shales/clays. The Bima Formation is overlying by Cenomanian transitional Yolde Formation and followed by the marine Turonian – Coniacian Dukul, Jessu, Sukuliye and Numanha (Shales) Formations (Sarki Yandoka *et al*., 2014). The Lamja Formation, a delta sequence overlies the Numanha (Shales) Formation (Fig. 2). The Dukul Formation consists of bedded shales and fossiliferous limestone (Carter *et al*., 1963). The formation was dated as Early to basal Middle Turonian. Based on micro-fauna, the formation was interpreted as deposition in littoral to open marine shelf environments.

3.1 METHODS

Fieldwork was conducted on the exposed outcrops of Dukul Formation. Sections were described and measured. The formation composed of shales and limestones. The limestones are grain supported and rich in bivalves and gastropods. Some sections consist of mainly shales with siltstone and limestone intercalations. The limestones, shales and siltstones are grey of dark grey in colour, the shales are weathered.

After lithostratigraphic sections description and measurements, ten (10) shale samples were collected and prepared for laboratory analyses. An X-ray Powder Diffraction (XRD) analysis was performed on the powdered sample using SIEMENS D5000 X-ray diffractometer. The minerals were identified from the diffractograms by referencing to the ICDD Powder Diffraction File. About 0.50g of each sample was prepared for non-destructive wavelength dispersive X-ray fluorescence spectrometer (PANalytical Axios mAX 4KW sequential XRF spectrometer).

About 0.50g of each sample was weighed in a teflon beaker and dried at 105oC vernight. 5ml of Nitric acid (HNO3) was slowly added and placed on a hotplate at 1500C until it reaches near dryness and allowed to cool. 10ml of hydrofluoric acid (HF) was slowly added followed by 4ml of perchloric acid (HClO4). The samples were decomposed on the hotplate at approximately 200^{0}C until it reaches near dryness with a crystalline mush. After cooling, the samples were digested with 10ml of 5M HNO3 in a fume hood. The solutions were diluted with deionized water to 50ml in a volumetric flask. Digested samples were diluted with ultimate pure water. Trace and rare earth elements were determined using Agilent Technologies 7500 Series Inductively-coupled plasma mass spectrometer (ICP-MS) analysis. Analytical results are presented in Tables 1 and 2. Lithostratigraphic sections and field photo is shown in Figures 3 and 4.

Table 1: *Oxides of major elements (wt.%) for shales of Dukul formation*

Sample ID	Trace elements (ppm)									
	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	P_2O_5	CaO	MgO	Na_2O	K_2O	K_2O/Na_2O
DKL6	**59.0**	**10.2**	**0.65**	**6.18**	**0.18**	**0.41**	**0.62**	**0.23**	**2.98**	12.9
DKL3b	49.8	11.4	0.64	6.62	0.23	0.37	0.58	0.27	3.21	11.8
DKL5	61.3	14.1	0.81	7.12	0.13	0.38	0.66	0.31	3.40	10.9
DKL1	52.9	11.5	0.68	6.32	0.15	0.56	0.62	0.28	3.11	11.2
DKL2	54.8	12.8	0.67	5.67	0.20	0.62	0.56	0.22	2.96	13.5
DKL3	59.0	10.2	0.65	6.18	0.18	0.41	0.62	0.23	2.98	12.9
Average	**54.4**	**12.7**	**0.75**	**6.40**	**0.21**	**0.74**	**0.61**	**0.25**	**3.1**	**12.5**

Table 2: *Trace elements concentration (PPM) for shales of Dukul formation*

Sample ID	Trace elements (ppm)												
	V	Ni	Cu	Cr	Sr	Ba	Rb	Ga	Co	Sc	Ni/Co	V/Sc	Sr/Ba
DKL6	**45.2**	**8.9**	**5.03**	**14.1**	**22.9**	**98.9**	**35.8**	**12.5**	1.5	**2.5**	**5.90**	**18.1**	**0.23**
DKL3b	67.7	7.9	6.30	16.4	31.6	118	42.4	18.2	1.3	4.2	6.07	16.1	0.27
DKL5	66.5	8.4	6.22	19.2	41.1	97.9	38.9	10.8	1.4	3.7	6.01	17.9	0.42
DKL1	43.6	11.2	6.11	12.6	19.0	119	37.1	16.4	2.2	2.1	5.01	20.7	0.16
DKL2	48.9	5.4	5.60	13.6	34.6	116	45.6	14.2	1.1	2.4	4.91	20.3	0.30
DKL3	45.2	8.9	5.03	14.1	22.9	98.9	35.8	12.5	1.5	2.5	5.90	18.1	0.23
Average	**53.5**	**9.2**	**5.4**	**15.0**	**26.1**	**114**	**36.3**	**14.6**	**1.68**	**3.08**	**5.54**	**17.7**	**0.23**

4.1 RESULTS AND DISCUSSION

4.1.1 Mineralogy and elemental composition

The analysed shale samples contain detrital and non-detrital minerals (Fig. 5) with quartz as the most dominant mineral as shown in XRD pyrograms. Other minerals are kaolinite, glauconite, montmorillonite, pyrite, illite, chlorite, hematite and calcite (Fig. 5). Glauconite is diagnostic of shallow marine shelf environment where sedimentation of terrigenous clastics and carbonates become very slow in an oxygen-deficient condition.

Major elements are dominated by SiO2 (av. 59.4%), Al2O3 (av. 12.7%), Fe2O3 (av. 6.4%), and K2O (av. 3.1%) while others (CaO%, MgO%, Na2O%, TiO2%, P2O5%, MnO %) have concentration of mostly <1.0%. The results of XRD results are compatible with the oxides of major elements (see Table 1). The high percentage of SiO2 agreed with the dominance of quartz-based on the XRD analysis (see Fig. 5). Aluminium (Al) is also enriched in kaolinite, while Potassium (K) is associated with illite or glauconite mineral. Titanium (Ti) has known association with terrestrial detritus in high energy environments and mostly originates from mixed clay assemblage. Diagenetic alteration of volcanic ashes leads to enrichment of Al2O3, CaO, Fe2O3 and P2O5 (Bhatia, 1983).

The concentration of MgO, MnO and TiO2 are unaffected (Spears and Rice, 1973). The shales samples consist of SiO2, Al2O3, TiO2, Fe2O3, MnO, MgO, CaO, Na2O, K2O and P2O5. SiO2 is the dominant constituent followed by Al2O3 and Fe2O3 (Table 1). The contents of Al2O3 and SiO2 always change in the reverse direction. SiO2 occurs in compositions of coarse sediments, whereas Al2O3 formed from the component of clay minerals, therefore, there is a correlation between them. TiO2, Al2O3, Fe2O3, MgO, CaO, Na2O and K2O decrease with increase in the SiO2 content.

Calcium (Ca) concentration is explained by calcium carbonate minerals, while magnesium (Mg) is related to particularly chlorite, as well as carbonates. High Iron (Fe) contents indicate the source from pyrite, hematite or glauconite (Bhatia and Crook, 1986). Trace element concentrations of the shale samples with several widely used geochemical ratios are listed in Table 2. The trace elements concentration shows Ba, V, Rb, Sr and Ni with average values of 14.0, 53.5, 16.3, 26.1 and 9.2 ppm respectively, while Cr, Ga and Cu have average values of 15.0, 15.6 and 5.4 ppm respectively (Table 2). Strontium (Sr) and Barium (Ba) are elements that differed in their geochemical behaviour in various sedimentary environments. Sr/Ba ratio is regarded as an indicator of paleo-salinity.

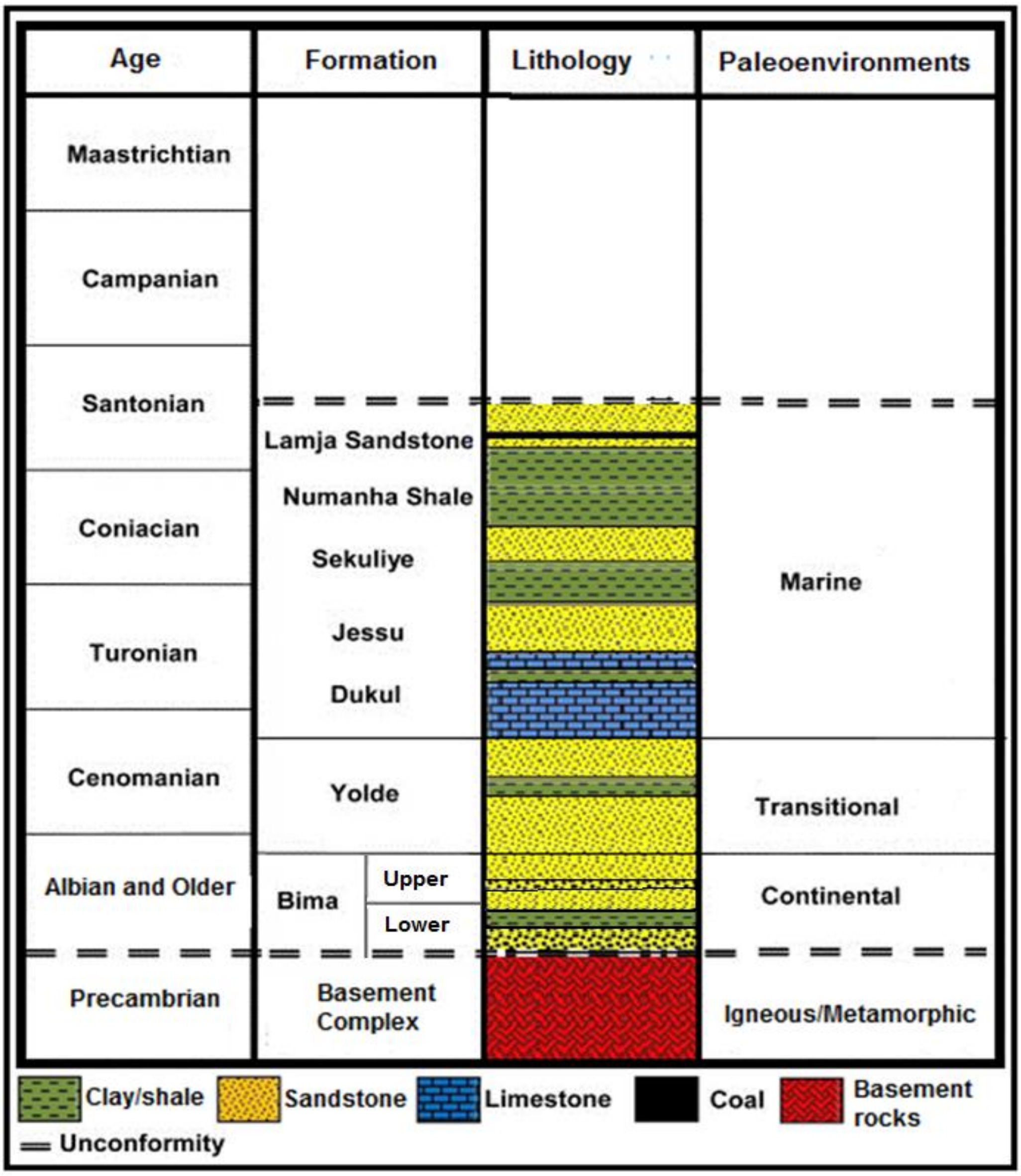

Figure 2: *Stratigraphy of Northern Benue trough*
Source: *After Sarki Yandoka, 2015)*

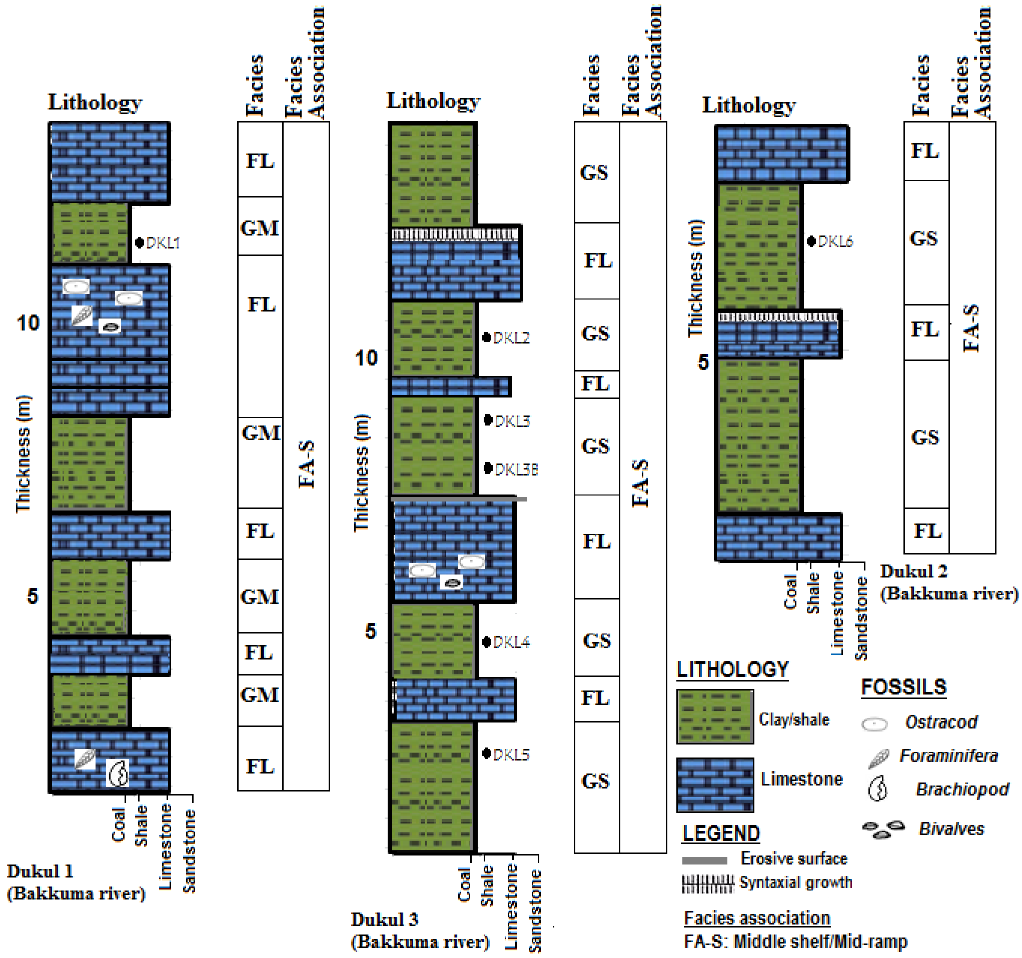

Figure 3: *Lithostratigraphic logs of Dukul formation from Yola Sub-basin*

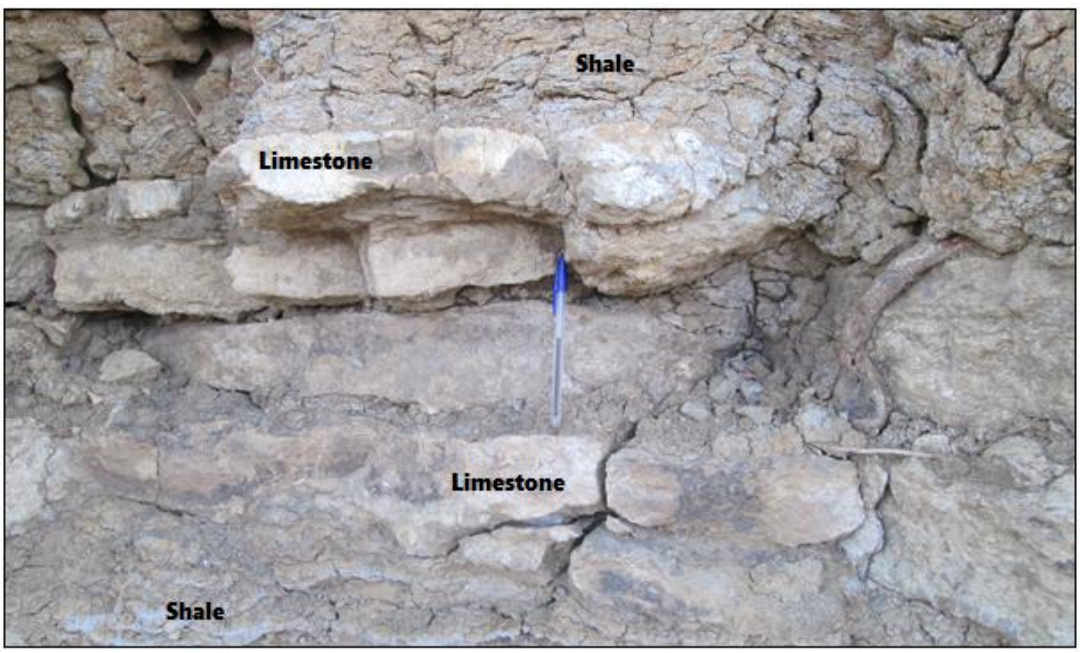

Figure 4: *Field photograph of Dukul Formation sediments at Bakkuma Rive*

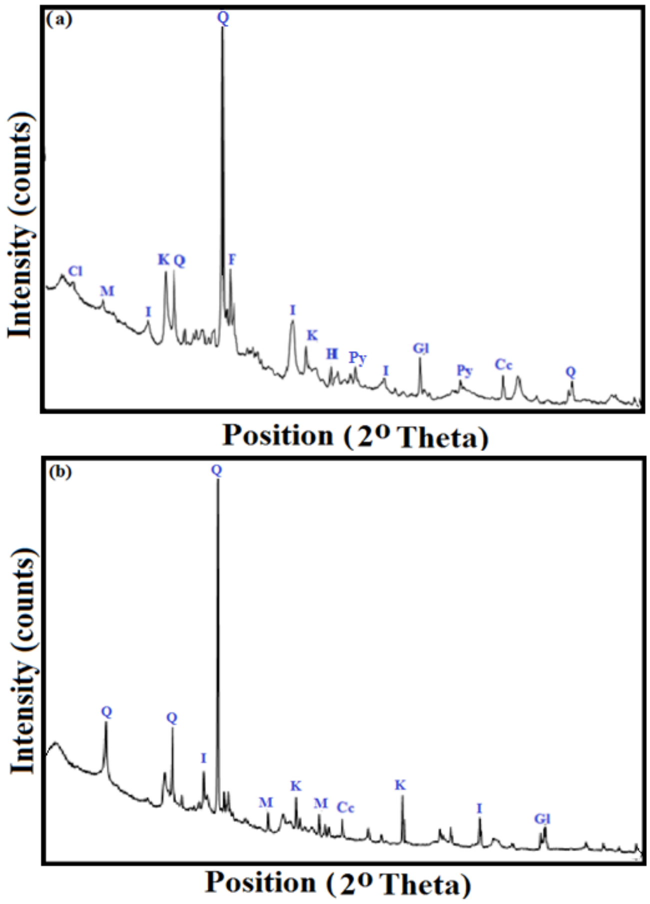

Figure 5: *X–ray diffractogram of the Yolde Formation shale showing the presence of Q-Quartz, F- Feldspar, Py-Pyrite, I-Illite, M-Montmorillonite, Cc-Calcium carbonate, K-Kaolinite, G-Glauconite, Cl-Chlorite*

High Sr/Ba ratio (avg. 0.67) indicates moderately saline water during deposition. The average ratio of Sr/Ba (0.23) may also indicate a significant marine influence. V is usually enriched in comparison with Ni in anoxic marine environments (Peters and Moldowan, 1993). Most trace elements are concentrated in mud-stone compared to fine-grained sandstone except for elements Sr, Zr, and Hf. The following trace and rare earth elements were detected from the samples; Co, Cr, Cu, Ni, V, Zn, Sr, Ba, Rb, Ga and Th (Table 2).

4.2 Paleodepositional environment condition

Palaeo-redox conditions during sedimentation can also be evaluated from trace elements data (see Table 2). Vanadium (V) and Nickel (Ni) are good indicators of redox conditions during deposition (Galarraga *et al*., 2008) but the relative proportions of V and Ni are controlled by the depositional environment. The ratio of V/Ni that is greater than 3 indicates deposition in a reducing environment, while the ratio of V/Ni ranging from 1.9 to 3 is indicating deposition under suboxic conditions (Galarraga *et al*. 2008). The concentration of vanadium (V) is higher than a nickel (Ni) in all the analysed shale samples (Table 2) with V/Ni ratio in the range of 1.90–2.54 indicate that the shales were deposited under suboxic conditions.

Standard ratios such as V/Ni and Sr/Ba are most commonly used as indicators for the redox conditions during deposition (Galarraga *et al.*, 2008). The samples intercalated with carbonate rocks and have the influence of marine conditions. This point of view is supported by a high concentration of elements (e.g., Ba, Sr, Rb and V) which are higher in seawater than freshwater. The relative proportions of V and Ni are controlled by the depositional environment. Cross plot of V and Ni supports suboxic condition (Fig. 6). V/Ni ratio greater than 3 indicates reducing environment, while V/Ni ratios ranging from 1.9-3 indicate deposition under relatively suboxic conditions with the precursor of mixed origin.

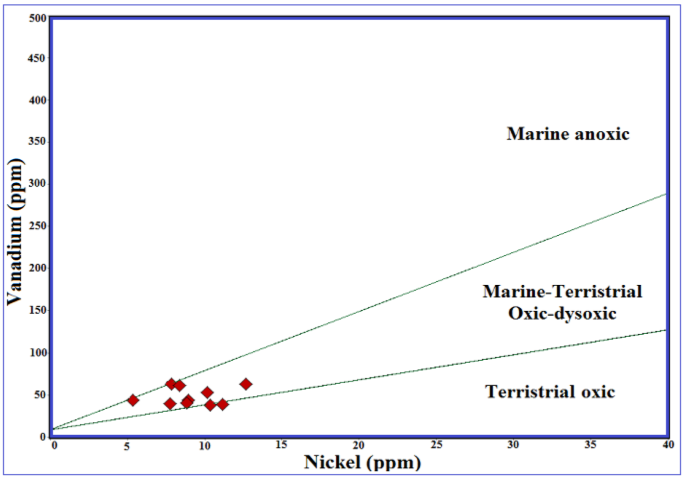

Figure 6: *Cross plots of Vanadium and Nickel showing paleodepositional condition*

The strontium (Sr) and Barium (Ba) differ in their geochemical behaviour in many sedimentary environments (Wang *et al.*, 2008). The ratio is regarded as an indicator of paleo-salinity. Therefore, high Sr/Ba ratio reflects high salinity, and low Sr/Ba ratio indicates low salinity. The Sr/Ba ratio of the analysed samples indicates a moderate saline water condition during the deposition of the sediments. Sr/Ba ratio and V/Ni ratio shows low salinity stratification and relatively suboxic conditions during deposition (Peters *et al.*, 2005; Peters and Moldowan, 1993).

4.3 Paleoclimatic condition

Paleoclimatic conditions during sedimentation of rocks can be evaluated from chemical analyses (Jacobson *et al.*, 2003; Suttner and Dutta, 1986). Suttner and Dutta (1986) proposed a binary SiO2 versus (Al_2O3+K2O + Na2O) diagram to severely restrict the paleoclimatic condition during sedimentation. The samples plot in the field of arid climatic conditions (Fig. 7); intermediate, indicating semi-arid/warm climatic condition (Roy and Roser, 2013; Ross and Bustin, 2009).

Aluminum (Al) and Gallium (Ga) are generally enriched in kaolinite, associated with a warm/humid climate (Ratcliffe *et al.* 2010). Potassium (K) and Rubidium (Rb) are associated with illite reflecting weak chemical weathering and related to dry or perhaps semi-arid climatic conditions (Ratcliffe, *et al.* 2010). However, most of the sediment-rich in illite should have low Ga/Rb and high K2O/Al2O3 ratios, whereas those that are rich in kaolinite will have high Ga/Rb and low K2O/Al2O3 ratios. The ratios support semiarid climatic conditions during deposition.

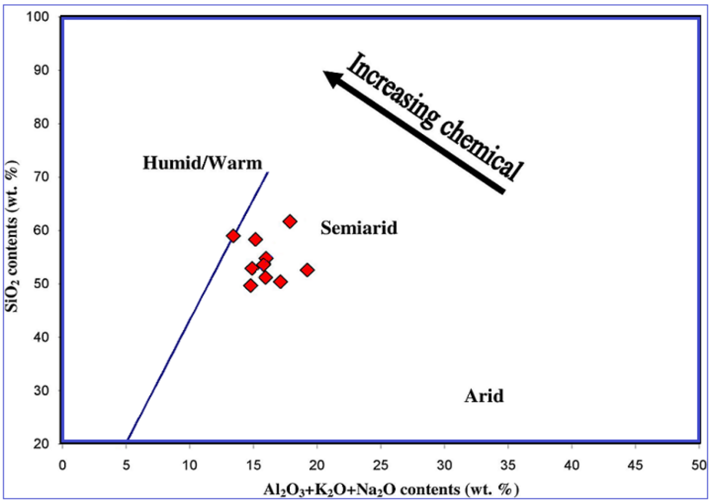

Figure 7: *Cross plot of binary SiO2 versus (Al2O3+K2O + Na2O) of shale samples*

4.4 Tectonic setting

Many classifications were used to discriminate various origins and tectonic settings of sediments (e.g. Roser and Korsch, 1986, 1988). Roser and Korsch (1986) used to log (K2O/Na2O) versus SiO2 (Fig. 8) to determine the tectonic setting of the source of terrigenous sedimentary rocks. SiO2 and K2O/Na2O increase from volcanic-arc to active continental margin to passive margin settings. The samples plot in the active continental margin field.

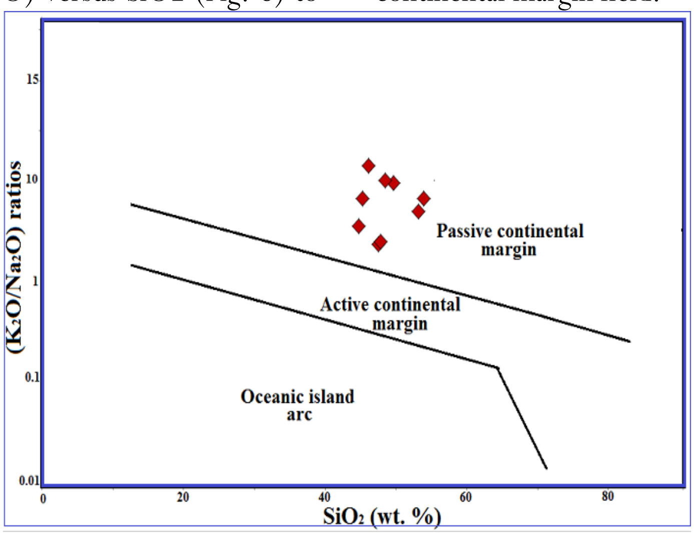

Figure 8: *Cross plot of Log (K2O/Na2O) versus SiO2 for the shale samples*

Scandium (Sc) is useful for provenance and tectonic setting determinations because of low mobility during sedimentary processes. These elements are transported into clastic sedimentary rocks and transferred quantitatively into the sedimentary record. Sc values in the analysed samples reflect a uniform continental provenance. The tectonic setting inferred for the provenance of the Dukul Formation is in agreement with the tectonic events witnessed in West and

Central African Rift System (WCARS) during the Cretaceous period.

5.1 CONCLUSIONS

Geochemical investigation of the Cretaceous sediments of Dukul Formation in the Yola Sub-basin, to infer paleoenvironment, paleoclimate and tectonic setting have revealed the following:

i. The dominant minerals are quartz followed by kaolinite, glauconite, montmorillonite, pyrite, illite, chlorite, hematite and calcite. The presence of glauconite also indicates deposition in shallow marine (shelf) environment,

ii. Major elements identified are SiO2 followed by Fe2O3, CaO, Al2O3, and TiO2. Their assemblages and modes of occurrence supported terrigenous and marine origin,

iii. Assessment of trace elements Sr, Ba, V, Ni, Co, Cr and their ratios shows a stratified water column with moderate salinity and suboxic bottom water conditions,

iv. The dominant clay minerals identified are kaolinite and illite as confirmed by elements; Al, K, Ga and Rb. The assemblages and modes of occurrence indicated semi-arid climatic conditions,

v. The binary plot of SiO2 versus (K2O/Na2O) revealed passive continental margin setting consistent with the tectonic events witnessed in the West and Central Africa during the Cretaceous period.

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