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Biomarker correlaton between shale, limestone, and sandstone of Yolde formaton, Northern Benue Trough

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ABSTRACT

The Yolde shales, limestone, and sandstone in the Gongola Sub-Basin, Northern Benue Trough, Nigeria, were both studied in the field and laboratory using the gas chromatography-mass spectrometry to expand our knowledge on their origin, relationship, thermal maturities, and paleo-depositional environment. Results obtained from the field studies suggest that the Yolde Formation was deposited between the marine and continental settings. Other properties of the transitional environment observed include bioturbation, high fossil content, cross beddings, and evidence of mud crack. Results from the laboratory analysis obtained show that the studied oils are all related and believed to have been sourced from a common petroleum source rock within the studied basin. The total ion current of the oils studied shows the presence of an unresolved complex mixture consistent with oils that have undergone biodegradation. This is because all the studied oils were extracted from outcrop samples. Trisnorhopanes thermal indicators of the studied oils are observed to fluctuate between 1.08 to 1.7 consistent with oil formed from petroleum source rocks that are in the oil window. Equivalent vitrinite reflectance for the studied samples varies roughly from 0.88 to 1 which infers oils generated from rocks within the oil window. This is consistent with the cross plot of sterane ratio C_{29} 20S/ (20S + 20R) versus C_{32} homohopane ratio (22S/22S + 22R), which also confirms that the studied oils are thermally matured and very close to the peak of oil generation. Cross-plot of n-alkanes/ isoprenoids shows the dominance of organic matter that originated from marine and then mixed environments. The Yolde Formation is known to have a maximum burial depth beyond 2,000 m in some parts of the Northern Benue Trough. This is therefore expected to find non-degraded oils associated with the deeply buried Yolde Formation.

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1. Introduction

Oil and gas make up about 95% of Nigeria's foreign earnings (BPE Report, 2006; Wikipedia Foundation inc., 2006.) and have hovered as the major supporter of its economy since it was first discovered in commercial quantity in 1956. There are inferences amongst petroleum geologists that the Benue Trough could be an excellent host for oil and gas as well as strata-bound and structurally controlled mineral deposits (Abubakar, 2014; Bata et al., 2018; Lar et al., 2018; Yandoka et al., 2017).

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The Benue Trough is filled with about 6,000 m of cretaceous sediments and volcanic rocks. In Nigeria, the Trough stretches for about 1,000 km in length and about 150 km in width. The Benue trough is subdivided into three parts, namely, southern, central, and northern (Fig. 1). There are no defined borders separating each of these parts from the others, but locations holding the depocenters have been well recorded (Idowu and Ekweozor, 1993; Nwajide, 1990; Obaje et al., 1999; Petters, 1982).

Petroleum is known to come about at a depth beyond 2,000 m and above 60°C in temperature where the transformation of kerogen into oil, and later gas, starts (Bata et al., 2015). The presence of oil stains at surface outcrop exposures in the Yolde Formation might perhaps provide a clue to the generation, migration, and entrapment of petroleum in the Yolde Formation; which serve as an inference to hydrocarbon investigation in the Northern Benue Trough of Nigeria.

In this report, Yolde shales, limestone, and sandstone in the Gongola Sub-Basin, Northern Benue Trough, Nigeria, were studied to expand our knowledge on their origin, relationship, thermal maturities, and the paleo-depositional environment through gas chromatography-mass spectrometry (GC-MS) fingerprints. These can be achieved by taking the superiority of biological compounds' efficacy in providing richer information needed to answer questions concerning the source, relationship, depositional environment, and thermal maturity of extracted oils than non-biomarker analysis. Thereby, providing more knowledge on the petroleum generation prospects in the Gongola sub-basin of the Northern Benue Trough.

2. Tectonic and Stratigraphic Setting

2.1. The Benue trough

Geographically, the Benue trough is roughly divided into Southern, Central, and Northern parts (Nwajide, 2013). There are no clearly definite lines of subdivision that exist to separate each part from the others, but there are famous towns/settlements hosting the depocenters of the individual parts that have been carefully documented. (Idowu and Ekweozor, 1993; Nwajide, 1990; Obaje et al., 1999; Petters, 1982).

2.2. The Northern Benue trough

The Northern Benue Trough is divided into two arms, namely, the Gongola Arm and the Yola arm. Some authors (Akande et al., 1998; Dike, 2002) consider the main (central) arm, Lau-Gombe, a sub-basin too, thus amounting to three sub-basins).

The Albian *Bima Formation is* unconformably overlying the Precambrian Basement and was deposited under continental environments which are fluvial, deltaic, and lacustrine environments. It consists of coarse to intermediate-grained sandstones with intercalations of carbonaceous clays, shales, and mudstones.

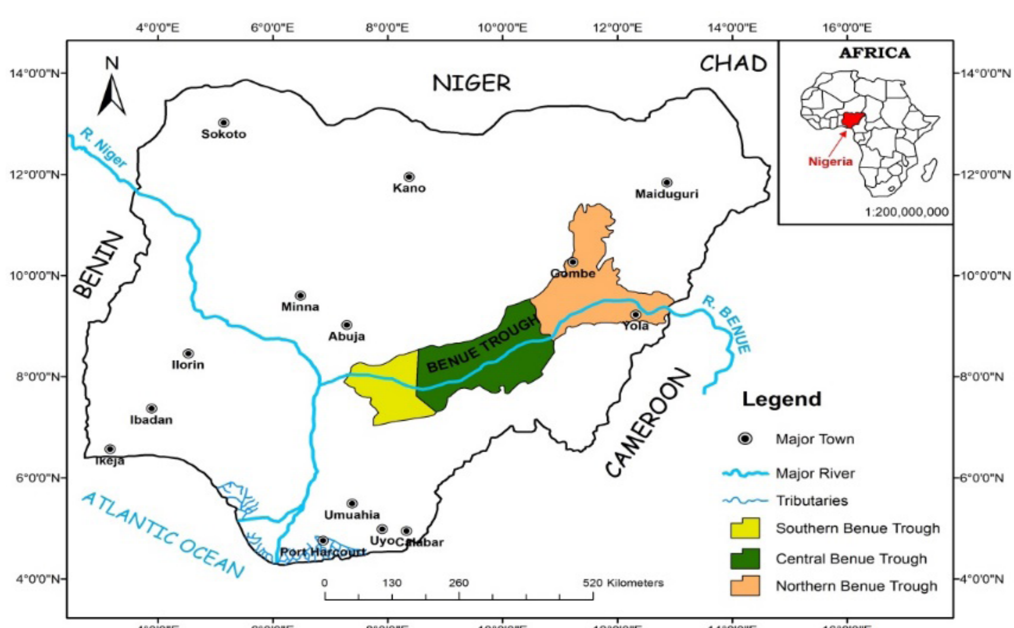

Figure 1. Map of Nigeria showing the major divisions of the Benue trough and study location (red box) (Bata et al., 2018).

Carter et al. (1963) subdivided the Bima Formation into Lower, Middle, and Upper Bima Members.

The Cenomanian in age Yolde Formation conformably overlies the Bima Formation, which marks the beginning of marine transgression. It is made up of fine to medium, moderately well-sorted feldspathic sandstones, an alternating sandy mudstone, thin shales, and fossiliferous limestone, with planar and trough cross-beds, ripple marks, and parallel laminations as the sedimentary structures. Type localities of the Yolde Formation are found along the stream channels of the Fika, Numan, and Pantami River in Gombe town and the village of Yolde.

The *Pindiga* Formations and maybe the younger *Fika Shale* conformably overlie the Yolde Formation (Obaje, 2009). The Formation designates a full marine incursion into the Northern Benue Trough during the Cenomanian–Turonian epoch times. The Formation consists of dark carbonaceous shales and limestones, with pale-colored limestones intercalation, and shales with minor sandstone. The Pindiga Formation was subsequently separated into five lithostratigraphic components. The Kanawa, Gulani, Deba Fulani, Dunbulwa, and Fika members.

The Gombe Formation is Maastrichtian in age, it sits on top of the Pindiga Formation and represents the youngest Cretaceous sediments in the Gongola Basin (Shettima et al., 2017). It is made up of intercalations of sands, shales, clays, coals, lignite, and coaly shale. It is visible around the Pantami River's bank and in many parts of Gombe town (Obaje, 2009; Onoduku et al., 2013).

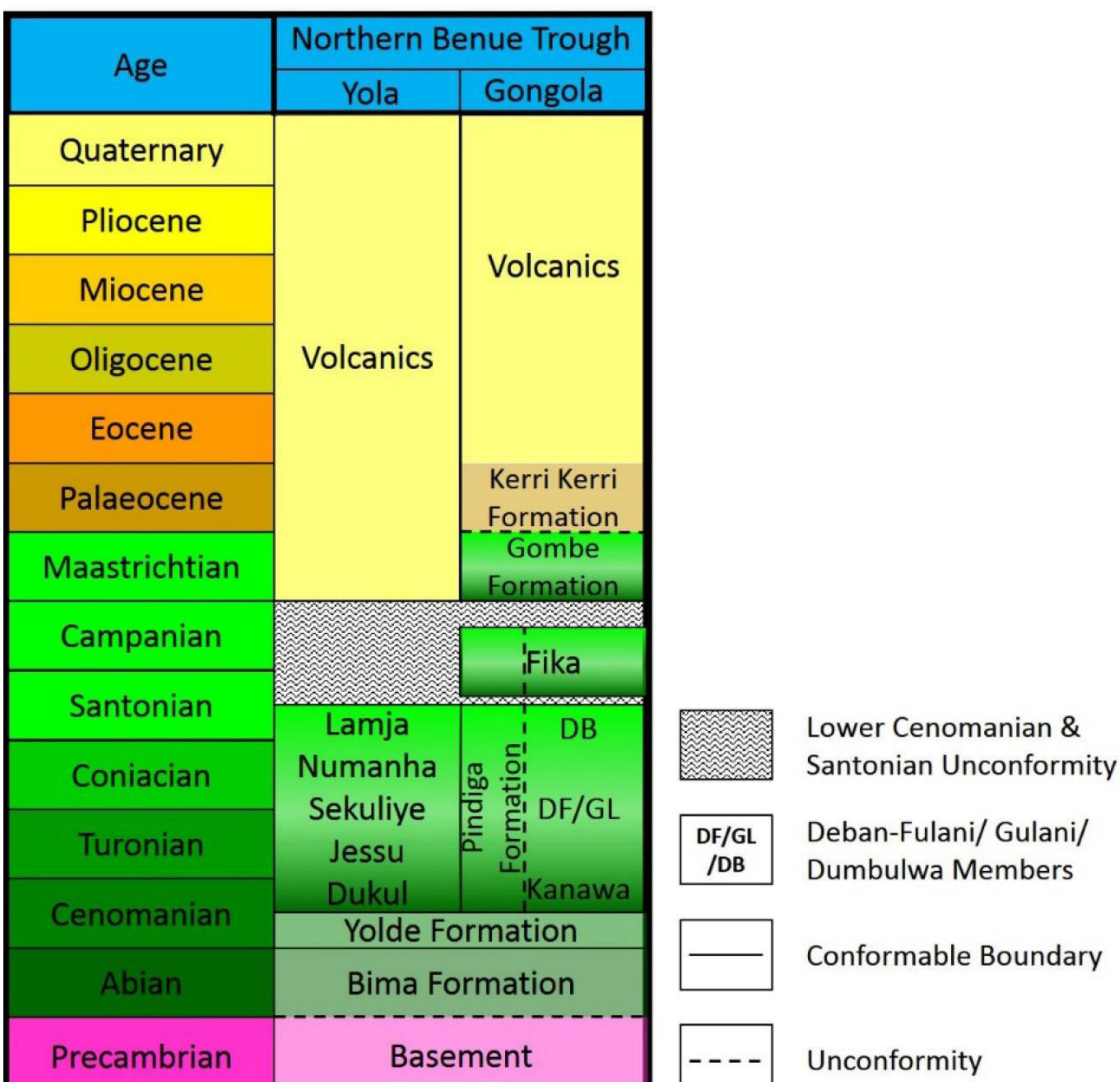

Figure 2. Stratigraphic successions in the Northern Benue trough (Lar et al., 2018).

The Tertiary Kerri-Kerri Formation of the Northern Benue Trough lies unconformably on the Gombe Formation (Dike, 1993). It is made up of claystone, whitish-gray sandstones, and siltstones. The outcrops are found in Gombe Aba, Duku, and Alkaleri (Obaje, 2009).

3. Methodology

During the fieldwork study, limestone, shale, and sandstone samples were acquired from exposures to the Yolde Formation. Fresh rock samples were obtained at separate intervals and tagged suitably (Plates I–IV). The samples were picked out and put through for organic geochemical analysis. Possible contaminated samples and near-surface deposits were carefully removed and set aside in sealed polyethylene bags and were made sure they were not too weathered to produce quality information. Samples taken for geochemical analyses were solvent-extracted using organic solvents 93:7 (*v*/*v*) dichloromethane, hexane, and methanol. The extracted oils were then divided into saturate, aromatics, and polar fractions engaging thin layer chromatography. The saturate fraction was then examined by gas GC-MS. The GC-MS analysis was carried out using Agilent 6890N GC equipped with a J & W DB-5 phase 50-m-long column that is connected to a 5,975 MSD and a quadrupole mass spectrometer working in selected-ion monitoring mode with ionization energy of 70 eV (Bata et al., 2015). The samples were manually introduced with a split/splitless injector working in splitless mode (purge 40 ml min^{-1} for 2 minutes). The temperature of the GC oven ranged between 80°C and 295°C. The GC oven was held at an initial temperature of 80°C for 2 minutes, further increased by 10°C $minute^{-1}$ for 8 minutes, then by 3°C $minute^{-1}$, and in the end at the highest temperature of 295°C for 10 minutes. Compounds were identified by matching retention times to well-distinguished materials that served as reference samples. The bulk and molecular composition of the oil extracted from the studied Yolde samples were obtained in order to determine the origin, relationship, thermal maturity, and paleo-depositional environment in the extracted oils. The discerned ions in the saturated hydrocarbons were m/z 85 for n-alkanes, m/z 191 for terpanes, and m/z 217 for steranes and diasterane.

4. Result and Discussion

This section presents the fieldwork data and laboratory analysis. The location of the Cretaceous oil samples in this study is shown in Figure 3. However, samples made-up of the organic-rich members of the Yolde Formation were obtained from the exposed outcrops in the study area. There were four representative samples, and one from each was picked and put through to organic geochemical analyses. The results include a geological map, field photographs, and biomarker data. These results are presented for comparing the oils occurring in the Yolde limestone, shale, and sandstone.

4.1. Field/hand specimen sample description

Figure 3 is the geologic map of the studied area showing the sampled localities. The oldest rocks occurring in the study area are the Precambrian basement rocks

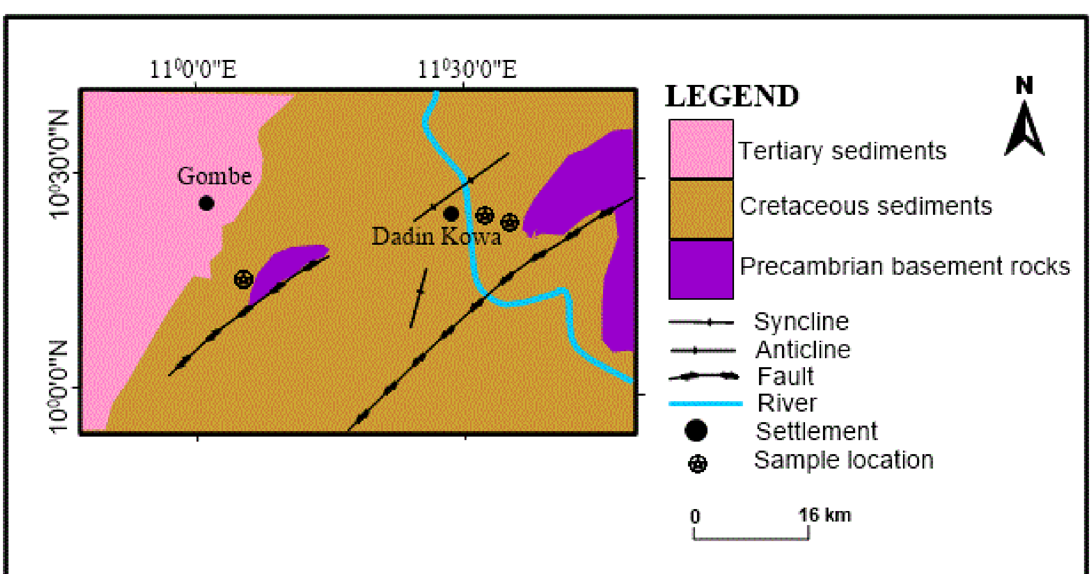

Figure 3. Geologic map of the study localities Gombe and Dadinkowa areas of the Gongola Sub-basin of the Northern Benue trough

which are unconformably overlain by Cretaceous and Tertiary sediments. This was followed by the deposition of the Cenomanian Yolde Formation consisting of sandstones, shale, and limestone (Akande et al., 2012). The Yolde Formation studied is discerned to be a key element of the potential petroleum system in the studied area due to the presence of oil and stains in the sandstones, shales, and limestones. Studied samples were collected from four localities within the Yolde Formation.

Plate I, Figures 4 and 5 are the field photograph and lithology of the studied section close to Biriyel town in the Northern Benue Trough. These sections are composed of medium to coarse-grain sandstone

Plate I. Photograph showing the sequence of Yolde sandstone with shale intercalation.

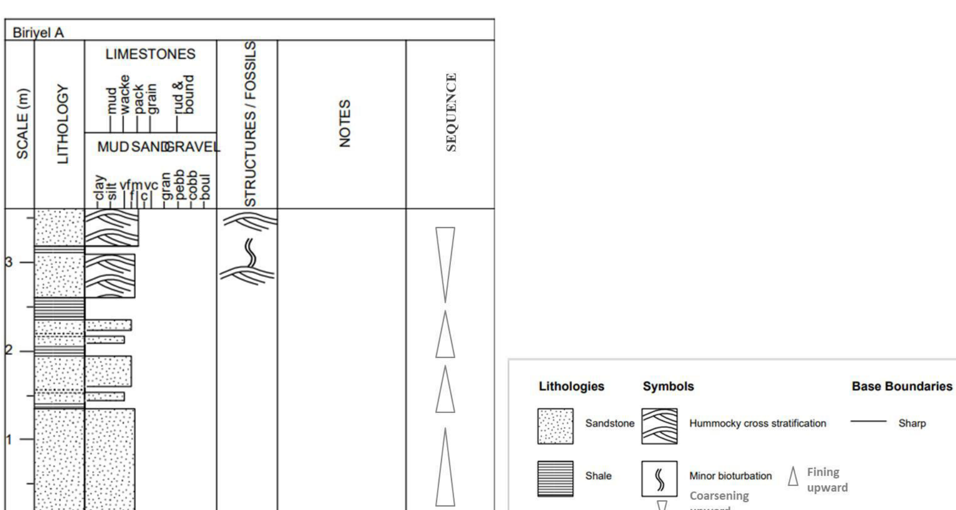

Figure 4. Lithostratigraphic section of the Yolde sandstone A.

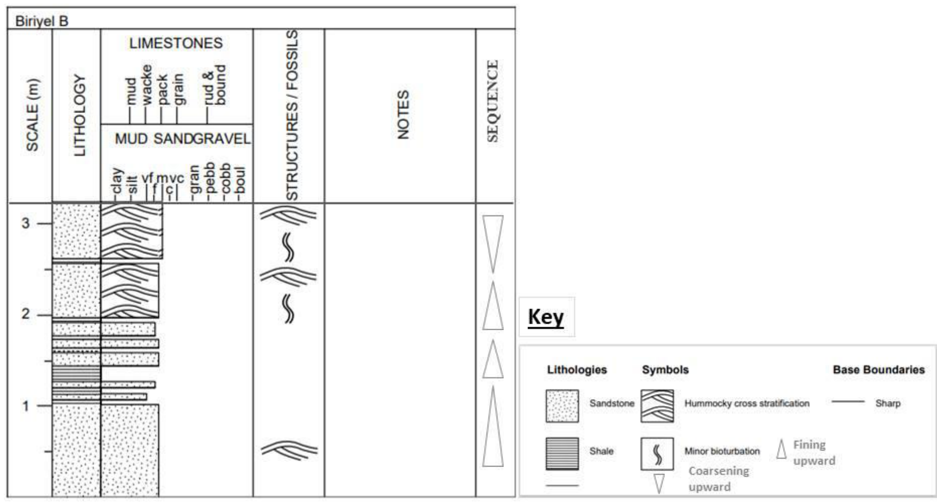

Figure 5. Lithostratigraphic section of the Yolde shale B.

Plate II. Photograph showing the oil-charged Yolde limestone.

interbedded with thin beds of shales with trough cross-bedding as the common sedimentary structure (Figs. 4 and 5). The section is more than 4 m thick and can be split into 3 fining upward sequences and 1 coarsening upward sequence (Figs. 4 and 5). There is no field evidence suggesting that the sandstones occurring at this locality are oil charged as seen in other studied localities.

Plate II is a limestone unit of the Yolde Formation occurring along the Biriyel-Dadin Kowa highway. These limestones are observed to be scattered on the surface covering a vast area. The limestones are rich in fossils, mostly ammonite suggesting Turonian age consistent with the age of the Yolde Formation in the Northern Benue Trough (Aliyu et al., 2017). The studied limestones are observed to be oil-charged

Plate III. Photograph showing the oil-charged Yolde sandstone.

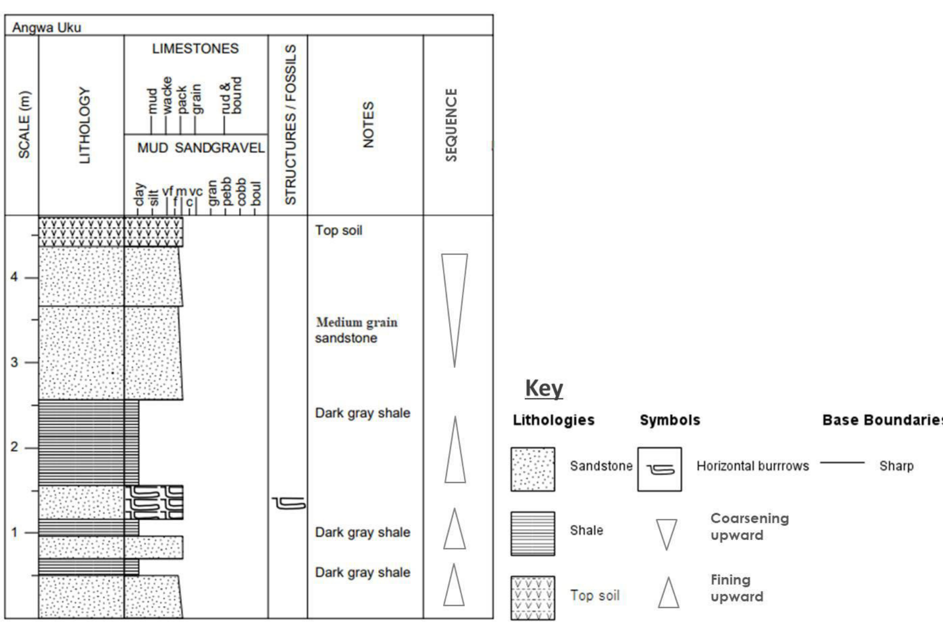

Figure 6. Lithostratigraphic section of the oil-charged Yolde sandstone.

(plate II) conforming to the existence of petroleum systems in the studied area.

Plate III and Figure 6 are the field photograph and lithology of the section studied at Angwa Uku locality in the Northern Benue Trough. The section is about 5 m thick, consisting of medium to coarse-grain sandstones that are interbedded with some dark to gray shales. The Angwa Uku section can be split into

Plate IV. Photograph showing the passive oil seepage on Yolde sandstone.

4 units consisting of 3 fining upward sequences and a coarsening upward sequence (Fig. 6). Unlike the sandstone section observed close to Biriyel (Plate 1), the sandstones occurring at the Angwa uku locality are observed to be black in color implying that they are oil charged, also suggesting the existence of a petroleum system in the Northern Benue Trough.

Plate IV shows evidence of a passive oil seepage associated with the Yolde Formation close to the Yaranduwa locality in the Northern Benue Trough. The passive oil seepage is observed to be on the surface and has darkened the associated sandstones. Oil seeps are good indicators of oil occurrence in the subsurface, and could reflect the migration of hydrocarbons (Bata et al., 2015).

4.2. The geochemical analyses

The geochemical data employed in this study is the biomarker acquired through the interpretations of GC-MS information developed on peak identification (Table 1). The compounds and their peaks identified from GC-MS data and the chemical compound they belong to are in Table 1. Oil extracted from the studied samples was analyzed using the GC-MS as described in the methodology section above. The total ion current (TIC) of the studied samples (Fig. 7) shows the presence of unresolved complex mixture (UCM) which is a characteristic of oils that have experienced biodegradation (Bata et al., 2015; Frysinger et al., 2003; Gouch et al., 1992; Ibrahim et al., 2022; Machel et al., 1995). This is expected as all the studied samples are outcrops samples. The grade of biodegradation can be observed to differ in the studied samples from a height of UCM (Fig. 7, Table 1). As seen in Figure 7 and Table 1, oil extracted from the studied limestone samples has a lower UCM height compared to oils extracted from the studied sandstone and shale samples. This indicates that oils linked with the studied limestone samples are less biodegraded compared to oils associated with the studied sandstone samples. This is expected as the sandstone samples will have more porosity and permeability to allow easy movement of bacteria in the rock compared to the limestones (Bata et al., 2015, 2018; Bata, 2016).

Trisnorhopane thermal maturity indicators of the studied oils are observed to fluctuate between 1.08 to 1.7 consistent with oil formed from petroleum source rocks that are in the oil window (Fig. 5, Table 1). Oils linked with the studied sandstone and shale samples have similar maturities implying a common history

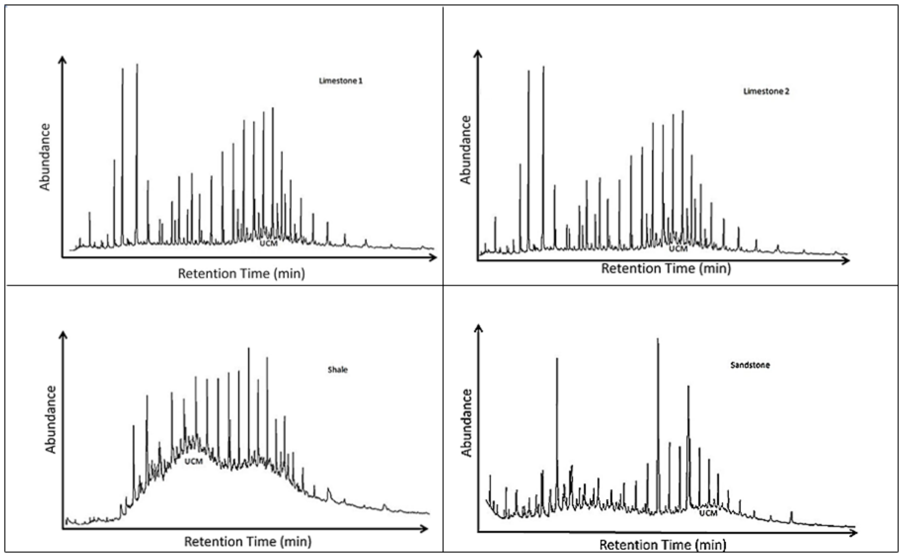

Figure 7. TIC fragmentogram of the saturate fraction of the studied Yolde oil samples showing UCM "hump" consistent with oils that have undergone biodegradation.

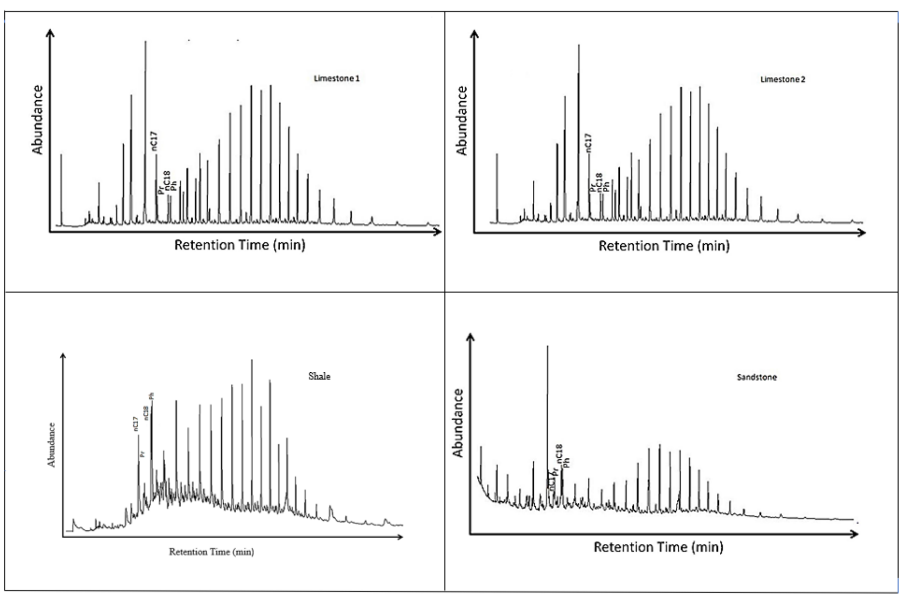

Figure 8. *m/z* 191 fragmentograms of the studied oil residue showing hopanes.

Table 1. Biomarker parameters from TIC, *m/z*191 and *m/z* 217 mass fragmentograms.

Oil sample	Pr/nC17	Ph/nC18	Pr/Ph	Ts/Tm	20S/(20S+20R)C_{29} Steranes	22S/(22S+22R)C_{32} Homohopanes	%C27	%C28	%C29	UCM height
Limestone 1	0.41	1.0	0.85	1.54	0.46	0.64	26.40	39.7	33.5	1.1
Limestone 2	0.43	0.97	1.0	1.7	0.46	0.63	26.8	39.6	33.6	1
Sandstone	1.45	0.95	0.84	1.08	0.47	0.55	33.8	30.7	35.3	1.2
Shale	0.78	0.52	1.72	1.08	0.48	0.52	33.3	35.0	31.7	5.3

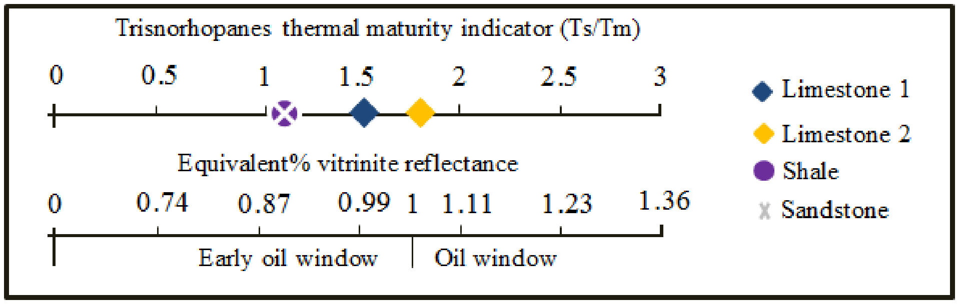

Figure 9. Trisnorhophane thermal maturity indicator (Ts:Tm ratio) for the studied Cretaceous oils samples showing the maturity levels of the oils with equivalent inferred vitrinite reflectance.

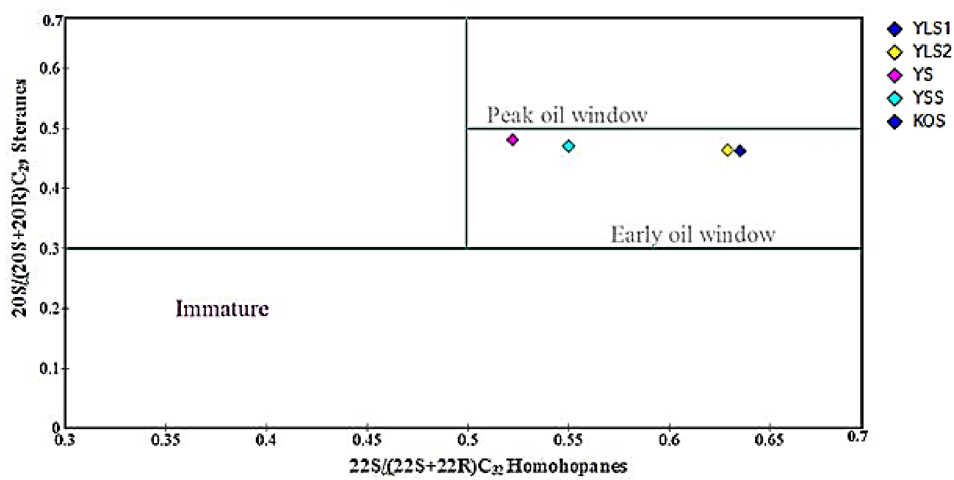

Figure 10. 20S/(20S + 20R) C_{29} steranes versus 22S/(22S + 22R) C_{32} homohopanes cross plot showing the thermal maturity of the organic matter in the analyzed samples. All the studied oils are within the early oil window.

regardless of the distance between the two sampled localities (Fig. 2). Differences observed in the trisnorhopane thermal indicator between oils extracted from the studied samples can be attributed to the effect of biodegradation. Biodegradation is known to affect Ts-Tm ratios (Bata, 2016; Peters et al., 2005). In such cases, the Tm is known to be faintly vulnerable to biodegradation relative to the Ts (Bata, 2016; Peters and Moldowan, 1993).

Equivalent vitrinite reflectance for the studied samples varies roughly from 0.88 to 1 which infers oils generated from rocks within the oil window (Fig. 5). This is consistent with the cross plot of sterane ratio C_{29} 20S/(20S + 20R) versus C_{32} homohopane ratio (22S/22S + 22R) presented in Figure 6 which also confirms that the studied oils are formed from the sources within the oil window and very close to the peak of oil generation.

Ternary plot presented in Figure 11 shows the relative amount of C_{27}, C_{28}, and C_{29} regular Steranes in the oils extracted from the studied samples. The studied samples are noticed to cluster on the ternary plot suggesting that the studied oils are related. Oils associated with the studied samples could have been sourced from shales associated with the Yolde Formation or other formations in the Northern Benue Trough. The limestones associated with Yolde Formation could be an important source of oil shows observed in the study area. However, the limestone beds in the study area are observed to be thin and sparsely scattered as oil shows within the studied area.

The ratio Pr/Ph (pristane/phytane) is a commonly used correlation standard that shows the depositional environment (Peters et al., 2005) of organic matter (Fig. 12). Pristane and phytane (tetramethyl hexadecane) come from the phytol side chain of chlorophyll, either under reducing conditions (phytane) or oxidizing conditions (pristane). Therefore, low Pr/Ph values (<0.6) specify anoxic environmental conditions,

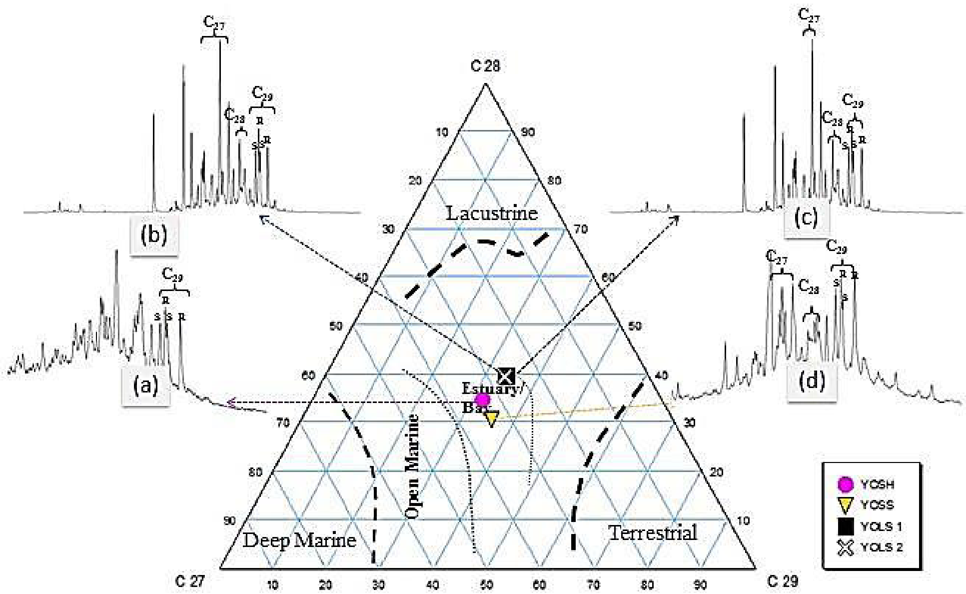

Figure 11. Ternary plot showing relative abundance of C_{27}, C_{28} and C_{29} regular Steranes in the studied samples. Note: clustering of the studied samples on the ternary implies oil from a common origin.

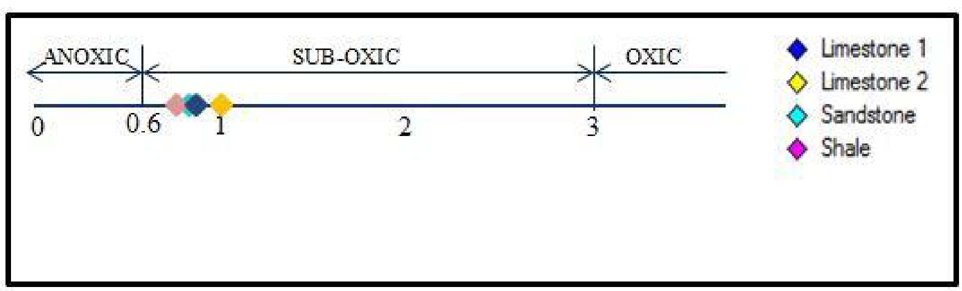

Figure 12. Pr/Ph ratios for the studied Yolde formation oils showing the environmental conditions of their deposition.

intermediary values (0.6–3) point to suboxic conditions and high values (>3) suggest oxic depositional environments (Peters et al., 2005). Pr/Ph ratios of the studied Yolde samples range from 0.84 to 1.75 consequently inferring oil source from sub-oxic depositional environments of organic matter. Generally, the Pr/Ph values of the four studied Yolde oils extracted are less than 2 and therefore point to aquatic environments of organic matter deposition including marine, fresh, and brackish water anoxic conditions (Lijmbach, 1975).

5. Summary and Conclusion

In this study, the oil geochemistry and molecular properties of oils associated with limestones, shale, and sandstones of the Yolde Formation were analyzed. Precisely, this report reveals the following:

i) The availability of a possible petroleum system allied with the Cretaceous Yolde Formation in Northern Benue Trough Nigeria.

ii) The exposure of the Yolde Formation at the surface and lacking seal in the study area exposed it to biodegradation.

iii) The studied oils originated from both marine and mixed organic matter deposited under sub-oxic conditions.

iv) The studied oils are inferred to have been derived from source rocks that are within the oil window almost at the peak of oil generation. Most likely the shales of the Yolde Formation are buried at a great depth.

v) The studied Yolde Formation is known to have a burial depth greater than 2,000 m in some parts of the studied basin. Oils related to the Yolde Formation that are deeply buried are likely to be of good quality.

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