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A Probable Connection between a Bitumen Sample from the Western Niger Delta and Isan Well Sample from the Lower Cretaceous (Neocomian) Shale

Selegha Abrakasa[1*], Koffi Eugene Kouadio[2,3], Franck-Hilaire Essiagne[2,3], Emma Laure Oura[4]

[1]Department of Geology, University of Port Harcourt, Rivers State, Nigeria
[2]Institut National Polytechnique Felix Houphouët-Boigny, Yamoussoukro, Côte d'Ivoire.
[3]Civil Engineering, Geoscience and Geographic Sciences Laboratory, Yamoussoukro, Cote d'Ivoire
[4]Center of Analysis and Research, PETROCI, Abidjan, Cote d'Ivoire
Email: *selegha.abrakasa@uniport.edu.ng

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Abstract

An earlier study of GC-MS analysis on bitumen samples from the Agbabu area in Western Niger Delta showed the significant presence of gammacerane and no Oleanane and is suggested to be generated from the Lower Cretaceous (Neocomian) source rock, however, twelve core samples were obtained from the geological survey of Nigeria for both optical geochemical and biomarker analysis, two of the samples from Isan-9 well indicated the presence of Botryococcus braunii a freshwater alga which is characteristic of a lacustrine lake type environment. The extracts from the same core showed m/z = 191 chromatogram with the significant presence of gammacerane which is diagnostic for stratified water columns and characterizes a typical lake environment. However, the m/z = 191 chromatogram also shows the presence of Oleanane which is diagnostic for vascular plant materials, this doesn't corroborate with that of the Ise-2 well. The Isan-9 well source rock is suggested to have occurred due to the evolving structural geology of the continental shelf after the onset of the rifting, whereby the dominant lake-type environment in the Early Cretaceous was accompanied by a sediment fill of continental runoffs containing vascular plant materials. This implies that an ancient lacustrine lake system was gradually modified as a result of sedimentary infill. And that the bitumen is sourced from the lower Cretaceous Neocomian source rocks.

Keywords

Amorphous, Botryococcus Braunii, Bitumen, Gammacerane, Neocomian, Niger Delta Basin, Oleanane

1. Introduction

Optical geochemistry entails the study of sedimentary organic matter obtained from shales that are insoluble in acids and some organic solvents with the use of a microscope otherwise also known as visual kerogen studies. This study has been a key aspect of petroleum geochemistry in derisking exploration and source rock assessment. Some advantages of optical geochemistry have been that a visual knowledge of the organic matter constituents is obtained, in addition, the preservation state and degree of thermal alteration are determined by direct optical examination. The hypothesis behind the validity of the optical geochemistry is that the constituents studied are integral parts of the rock matrix and adequately represent the rock in terms of its organic precursors, the environment of deposition, and thermal maturation [1].

The fate of organic matter in the matrix of source rock includes diagenetic processes which terminate with metagenesis, a post-maturation stage, where the organic matter is nearly stripped of hydrogen and thus becomes graphite (**Figure 1**). It commences with diagenesis which involves decarboxylation, hydrolysis, polymerization, and condensation within the organic precursor organism, modifying the functional groups of lipids and fats. The formation of kerogen is during catagenesis, which further disintegrates and degenerates to generate petroleum/crude oils.

The generated crude oils bear biomarker which is sometimes known as chemical fossils. The study of extracted sedimentary organic matter by transmitted white light microscopy is known as palynology, it can be applied to organic matter of any age and at any stage of diagenesis or maturation. The greater appreciation of the 3D morphology of the organic matter and the view through translucent particles ranks it as a reliable and cost-effective method for maturity and source rock potential studies [1].

The optical description and classification of sedimentary organic matter (kerogen) covers aspects such as its origin, structure, morphology optical properties (degree of reflectance), geochemical composition, and preservation state [1].

Biomarker geochemistry entails using mass chromatographic signatures to delineate the genetic characteristics of oils and source rocks. These signatures correspond to precursor compounds in the precursor organisms that undergo diagenetic alteration without completely modifying their functional groups. The signatures adequately represent the source as well.

This present study aims to combine optical geochemical and biomarker analysis to determine the possible link between the bitumen from the western Niger Delta and the shale of the Isan well sample from the Lower Cretaceous (Neocomian).

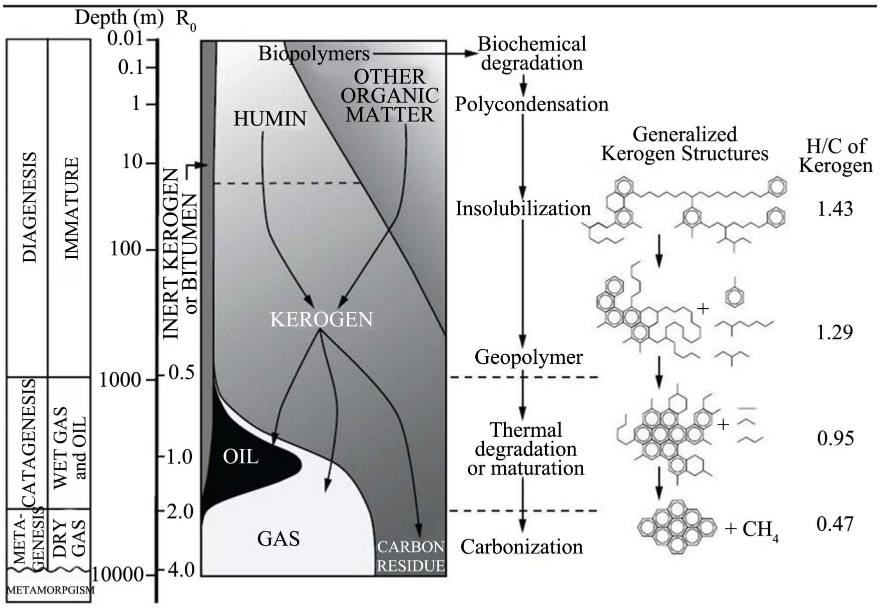

Figure 1. The fate of sedimentary organic matter through the various stages of diagenesis and transformation profile.

2. Niger Delta Basin

The sedimentary basin of the Niger Delta covers an area of about 256,000 km^2, it is one of the most prolific petroleum provinces in the world, it is located in Southern Nigeria [2].

It is bounded to the south by the Gulf of Guinea and the north by older tectonic elements (Cretaceous) including the Anambra Basin, the Abakaliki rising, and the Afikpo syncline, and to the east and west by the Cameroon volcanic line and the Dahomey basin respectively. The Niger Delta Basin commenced forming in the Late Cretaceous when the African plate separated from the South American plate [3]. The Niger Delta Basin consists of three main lithostratigraphic units of Cretaceous to Holocene origin. These units represent the prograding depositional environments which are distinguished mainly based on shale-sand ratio sand are continental, transitional, and marine environments. This Tertiary sequence in the Niger Delta consists of three formations that are locally designated in ascending order (from the bottom) the Akata Formation, Agbada Formation, and Benin Formation [4].

Petroleum system studies of the Niger Delta basin proposes three petroleum system, these are the Lower Cretaceous (lacustrine) petroleum system, the Upper Cretaceous to Lower Paleocene (marine) petroleum System, and the Akata-Agbada (deltaic) tertiary petroleum System. The Akata-Agbada (deltaic) tertiary petroleum System is the most active. Studies have established that seepages of bitumen from the Western Niger Delta Basin tarsand system bear similar geochemical characteristics with the Neocomian source rock of the Lower Cretaceous band of rocks in Ise-2 Well. Botryococcus braunii which is usually abundant in semi-permanent ponds, lakes, and slow-flowing and lacustrine regimes [1], has been identified in samples of rock from the shale interval in Ise-2 Well. In addition, extracts from the source rock samples in Ise-2 Well bear similar geochemical characteristics to the oils from the Lower Congo basin that have been derived from Lower Cretaceous Lacustrine Bucomazi source rocks. Some biomarker analysis of the bitumen samples from open pit mines of Agbabu, Loda and Petu, all within the Western Niger Delta showed the presence of gammacerane (**Figure 2** and **Figure 3**) which is a diagnostic marker for stratified water column, which is also a basic characteristic of lake type (lacustrine) environment. These mass chromatograms (**Figure 2** and **Figure 3**) do not indicate the presence of vascular plant materials as the precursor organic matter, there is no oleanane in the mass chromatogram. This observation may suggest some linkage of the bitumen to the Lower Cretaceous Neocomian source rocks.

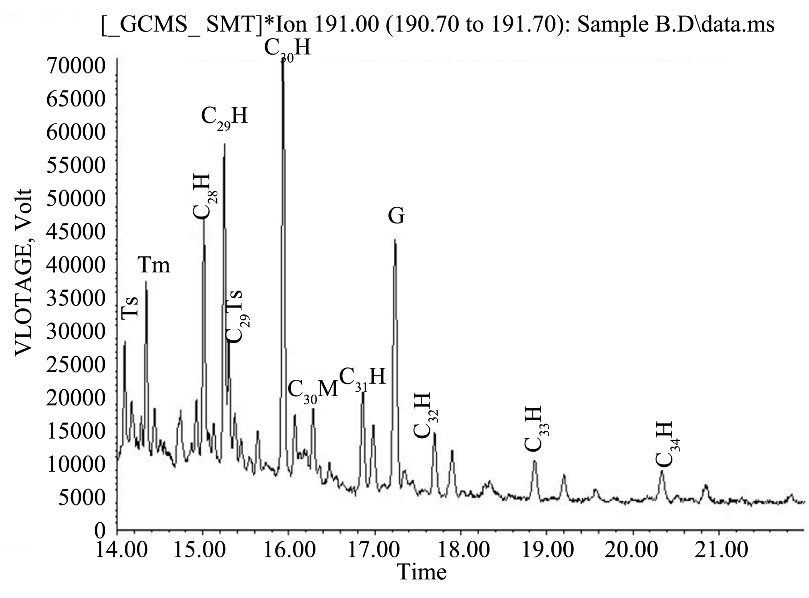

Figure 2. M/Z = 191 Mass Chromatogram of bitumen at Agbabu.

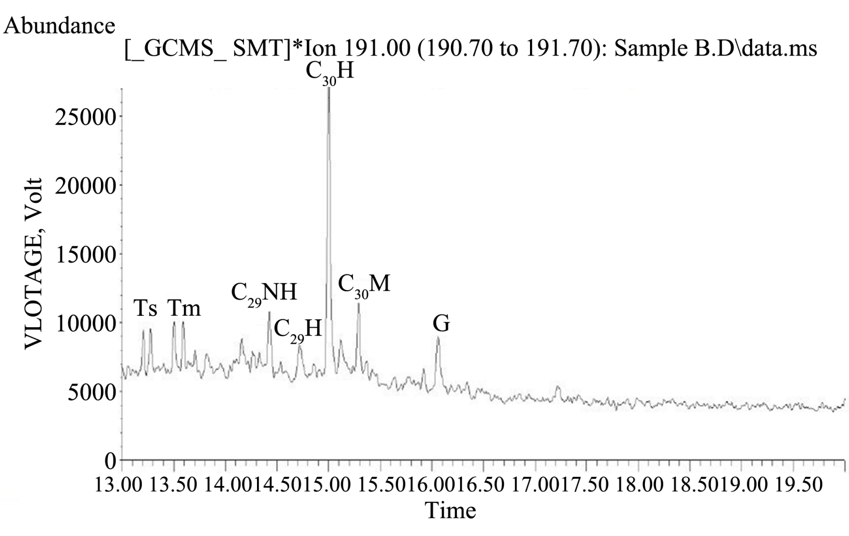

Figure 3. M/Z = 191 Mass Chromatogram of bitumen at Loda.

Agbabu, a town in Ondo State of Nigeria and is known as the home of bitumen, some outcrops bear features of bitumen seeping out from minor fractures (**Figure 4**), subsequently, a well has been drilled to confirm the deposit of bitumen (**Figure 5**). The hypothesis on which this study rest is that entrapment of migrated oils into the reservoir can only occur if there is a necessary configuration to trap the oil while the void space serves as the storage space, however, where the timing is wrong and there is not trapping mechanism, the migrating oils will subsequently get to the surface via grading sandstone bodies which will eventually be impregnated by the oil within the matrix of the extensive sandstone body.

The petroleum system of the Foreland Basin (**Figure 6**) can be used as a model to explain the circumstances that lead to the occurrence of bitumen and tarsand on the surface of grading out reservoir sand body, indicating the absence of a trapping configuration in place that could have been responsible for holding back the migrating petroleum thereby forming an accumulation. But rather migrates out to the surface impregnating the sandstone matrix and possibly seeping out from fractures and faults and over time getting biodegraded.

Figure 4. Picture of bitumen seeping out from fractures on an outcrop at Agbabu.

Figure 5. Picture of the bitumen well casing at Agbabu.

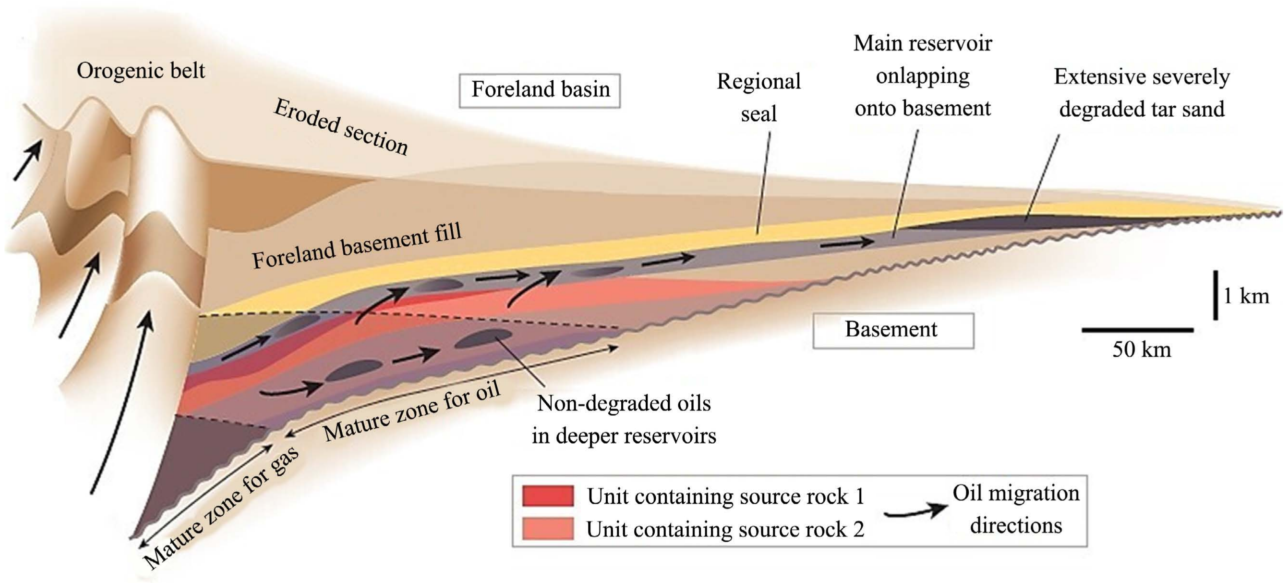

Figure 6. Foreland basin petroleum systems and petroleum migration profile.

3. Materials and Methods

Twelve (12) core samples of eleven (11) wells, in the onshore Niger Delta basin, were obtained from the geological surveys of Nigeria Kaduna. The samples were subjected to treatment with 37% hydrochloric acid (HCl) and 70% hydrofluoric acid (HF) for the removal of carbonates and silicate minerals. Kerogen was isolated by washing and mounted on a glass slide for analysis under transmitted white and fluorescence light for spore coloration index and kerogen type description, to estimate the relative abundances of vitrinites, inertinites, Amorphous Organic Matter (AOM), and maturity.

The analysis was carried out in the laboratory of PETROCI (Société National d' Opération Pétrolière de la Cote d'Ivoire), using a Transmitted Light and Fluorescent Light Microscope (Axioskop 40 FL).

The rock samples were washed with distilled and deionized water and air-dried prior to grinding. The samples were powdered to a particle size of about 425 μm or less. Approximately 50 g of each sample was finely crushed, and using a mixture (50:50 v/v) of dichloromethane (CH_2Cl_2) and methanol (CH_3OH) Soxhlet extraction was performed. The purpose of extraction is to recover the extractable organic matter (EOM). In Soxhlet extraction, the solvent is vaporized from a flask, passes upward through the side arm of the apparatus, condenses, and drips down into a cellulose thimble containing the powdered sample within the Soxhlet. The hot solvent extracts the soluble organic matter from the sample and is periodically recycled into the solvent reservoir flask via a siphon. Several cycles of this process are required to ensure complete extraction.

The fractions of bitumen obtained after Soxhlet extraction are dissolved in *n*-hexane and analyzed using gas chromatography-mass spectrometry (GCMS). The GC-MS analysis is performed on Perkin Elmer GC/MS Clarus 500. Chromatograms were acquired in scanning: 35 - 700 molecular weight and selected-ion-monitored (SIM) for compound identification and integration. The distribution of organic compounds in the bitumen extracts is monitored by fragmentograms of *n*-alkane (m/z 85), terpanes and hopanes (m/z 191), and steranes (m/z 217/218). The relative concentrations of particular compounds were calculated from peak areas.

Biomarkers were identified by comparison with the previously published library.

4. Results and Discussion

The results of the optical geochemistry (transmitted white light microscopy) indicate evidence of vascular plant materials for all the samples except the sample from Isan-9 well at both 6760 ft and 8680 ft.

The Isan-9 well results are presented in **Figure 7** and **Figure 8**, as could be observed, present in the images are circular sections such as cell walls, which infers structured cell wall, it exists as a discrete individual entity, with no presence of a fragmentary particle is observed. Thus, belongs to the palynomorph group.

The irregular globular colonies with several lobes which are like cauliflowers heads, that are glossy yellow to orange-brown with strong fluorescence in appearance indicate Botryococcus in the sediment.

Isan-9 Well (8680 ft) also showed the presence of a seed-like entity with a Y-shape and is identified as a spore, these are organic matter in the matrix of the rock that consists of the Isan-9 well shales. These organic matters are characteristics of deposits in lake-type (lacustrine) environments.

The biomarker analysis of the extracts from the Isan-9 well at 6760 ft and 8680 ft was performed; the result is indicated in **Figure 9** and **Figure 10** for M/Z = 85 and **Figure 11** and **Figure 12** for M/Z = 191 chromatograms. **Figure 11** and **Figure 12** indicate the presence of gammacerane in the samples, gammacerane is

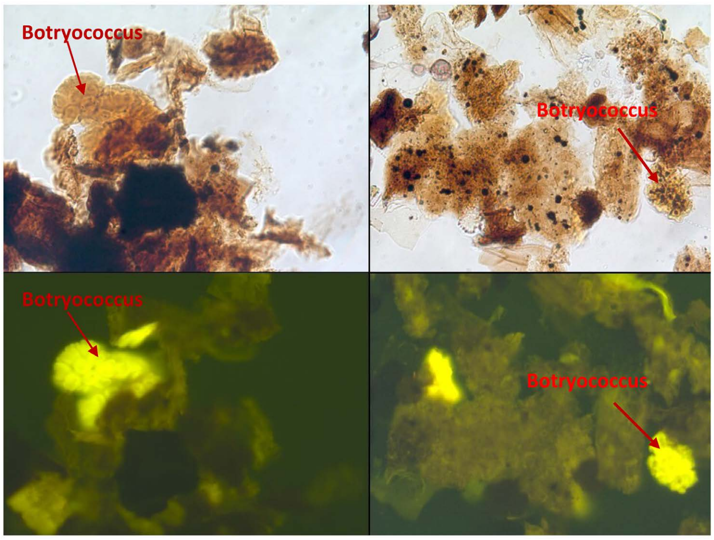

Figure 7. Photomicrographs of isolated organic matter in Isan 9 Well (6760 ft) rocks.

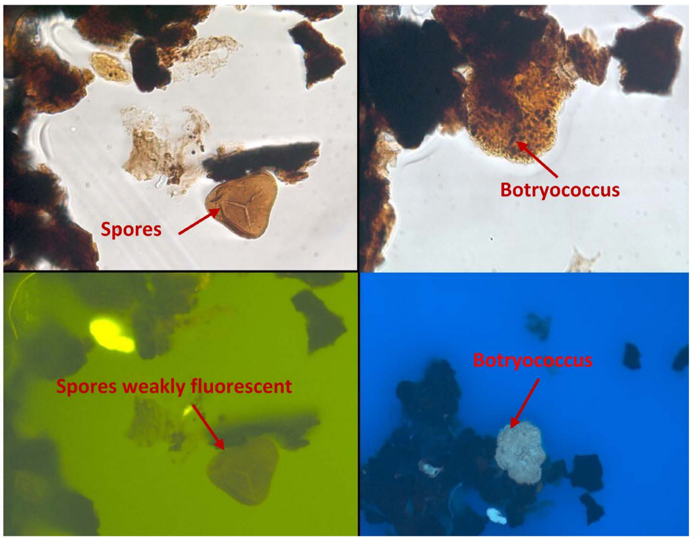

Figure 8. Photomicrographs of isolated organic matter in Isan 9 Well (8680 ft) rocks.

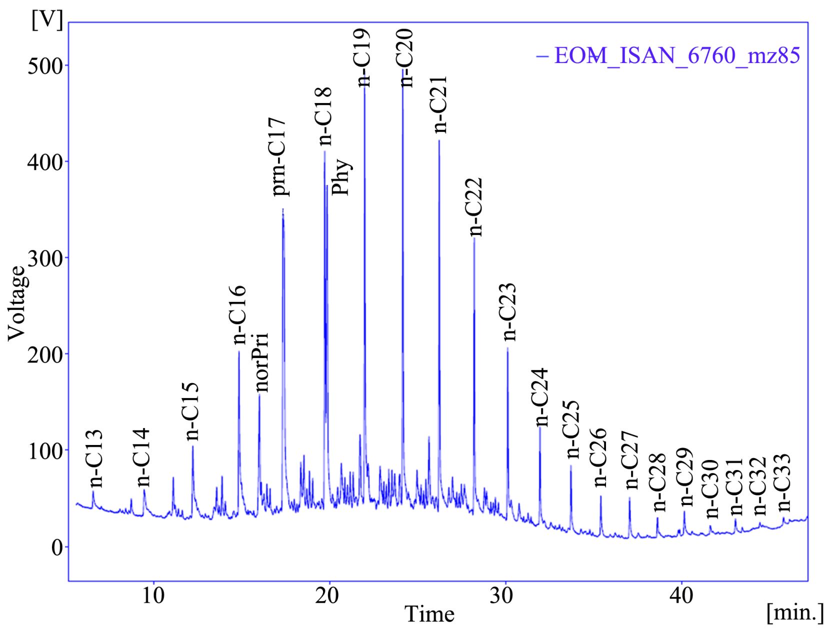

Figure 9. M/Z = 85 Mass chromatogram for Isan 9 Well (6760 ft) extract.

diagnostic for stratified water columns which is a common characteristic of a lacustrine environment.

The Isan-9 Well also showed the presence of Botryococcus (**Figure 7** and **Figure 8**), this observation also indicates the precursor organic source as lacustrine. However, the Isan-9 well extracts showed the presence of gammacerane (**Figure 11** and **Figure 12** [red arrow]) which is a diagnostic for stratified water column, a basic characteristic of freshwater lake environment and basin of restricted circulation on continental shelves [5]. In addition, the presence of Oleanane (**Figure 11** and **Figure 12**) is also observed this could be due to the evolving structural geology of the continental shelf after the onset of the rifting.

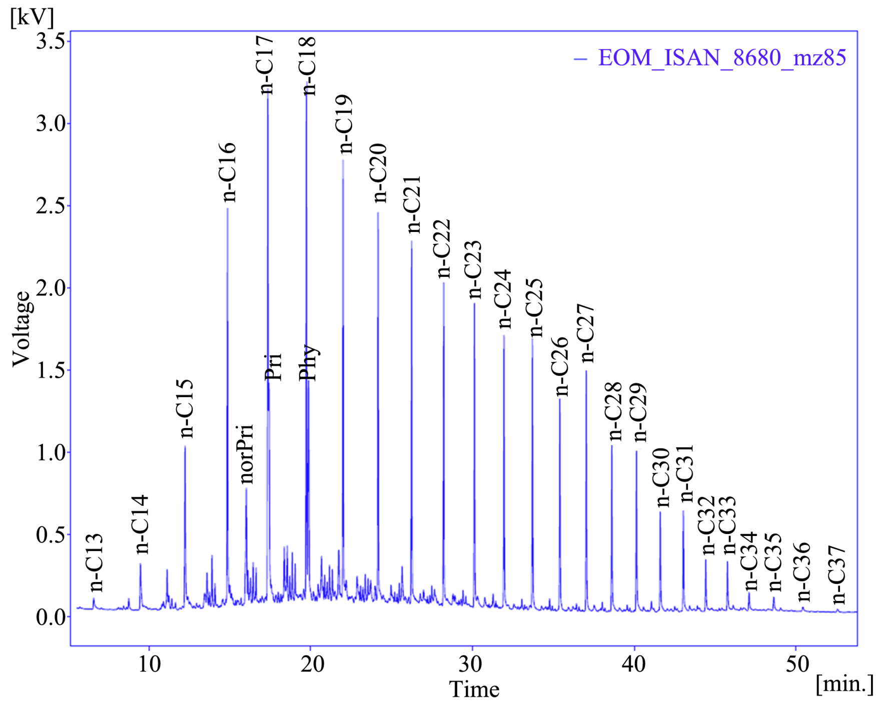

Figure 10. M/Z = 85 Mass Chromatogram for Isan 9 Well (8680 ft) extract.

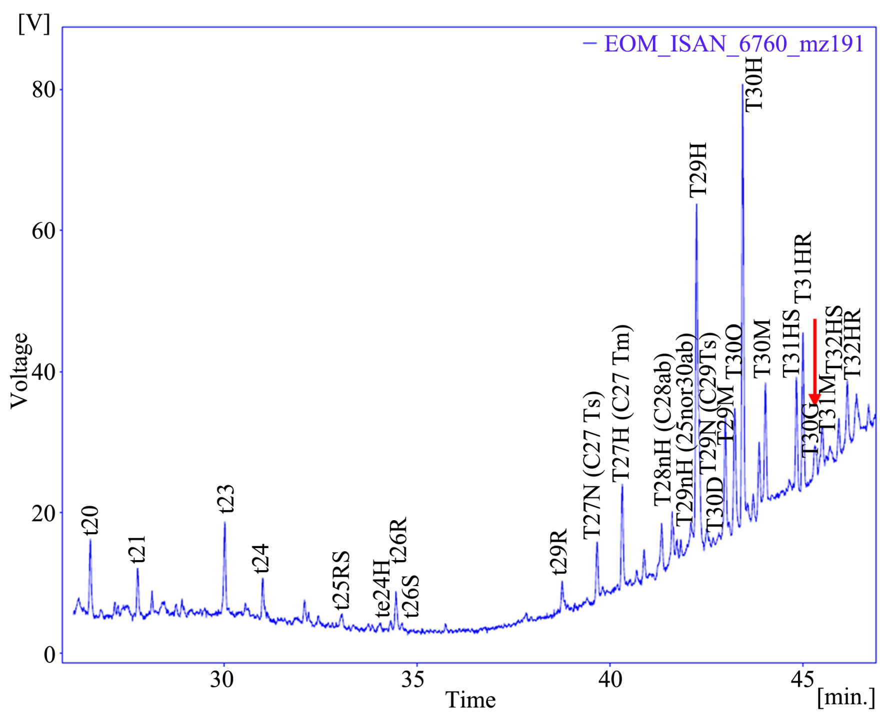

Figure 11. M/Z = 191 Mass Chromatogram for Isan 9 Well (6760 ft) extract.

The dominant lake-type environment in the Early/Lower Cretaceous was also accompanied by the onset of the presence of flowering plants in the Middle Cretaceous [6], hence potential continental runoffs could contain vascular plant materials which go into.

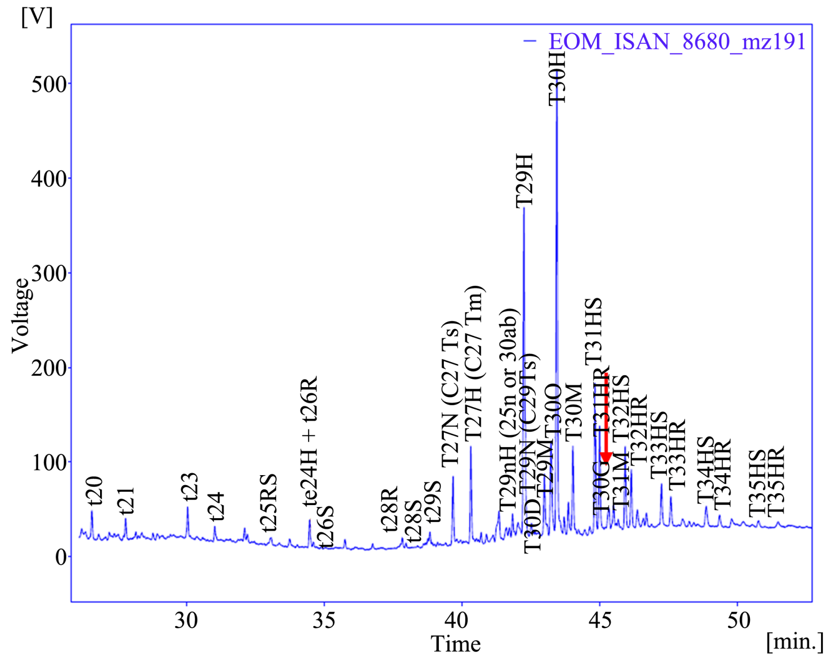

Figure 12. M/Z = 191 Mass Chromatogram for Isan 9 Well (8680 ft) extract.

The lake system and this explains the Oleanane signature. The Isan-9 Well is located in the same geographical region as the Ise-2 Well, geological they are located within the same geological region covered by the Lower Cretaceous Petroleum system [6].

The M/Z = 191 mass chromatogram for the bitumen indicates the absence of Oleanane, which is diagnostic for deltaic organic matter sourced oils. However, a significant presence of gammacerane is observed, characteristic of the stratified water column and common in lacustrine environments. This implies that the bitumen samples analyzed were sourced from the Lower Cretaceous lacustrine source rock. Prior to this time, it was suggested that the bitumen could have been sourced from the Upper Cretaceous Marine shales. But the molecular marker signals from the GC-MS analysis indicated that the most plausible source is lacustrine (**Figure 2** and **Figure 3**) not marine.

The structural evolution of the Niger Delta could be employed to explain the presence of Botryococcus in the shales of Agbada formation. It is suggested that an ancient lacustrine lake system was gradually modified as a result of sedimentary infill.

The asymmetric nature of the petroleum system and its potential is a result of the asymmetric nature of the rifting along the Southern Atlantic Continental Margin which is also reflected in the differences in the width of the continental crust, where the wide extended crust occurs in the North and the narrow band is present in the South.

This difference influenced the mechanism of subsidence, basin fill, and thermal history [6].

The initial system after the rifting was fresh to brackish to saline lacustrine water conditions, but each of the lacustrine basins that were established was filled as a consequence of basin shallowing through in-filling differently.

Rifting in the area of the Niger Delta Basin commenced in the Late Jurassic and continued into the Middle Cretaceous and diminished in the Late Cretaceous when the early flowering plants came into existence at the same time with gravity tectonics and slope instability due to the absence of lateral basinward support for under compacted delta slope clays [6]. It is possible that as the parts of the basin were transiting from lacustrine to marine it was accompanied by continental runoffs supplying vascular plant material to the basin. Hence the complex result as shown by the M/Z = 191 mass chromatogram with the presence of gammacerane and oleanane depicts a Lacustrine-Terrigenous source rock as the organic precursor.

Ola *et al.* (2019) suggested that the loading of the Lower to Middle Cretaceous sediment probably took place in a rather restricted graben in the Nigerian Sector of the basin [7]. The restricted graben probably corresponds to the portion of the basin, where rifting initially took place and accommodation was created for sediment accumulation.

Haack *et al.* (2000) stated that the Lower Cretaceous Petroleum System, which is characterized by the lacustrine source rock that occurs in the NorthWestern part of the Delta, a careful examination of the geographical extents of Lower Cretaceous and Tertiary Petroleum systems, unravels the fact that there is an overlap of the geographical extent of both systems [8]. This may imply an overlap of the depositional sequences. The Ise formation which is a Neocomian source rock has exposures at Ode-Remo on the Lagos-Ibadan expressway.

The presence of the Lower Cretaceous petroleum system in the Dahomey Basin has been observed and has been suggested to extend to the North East of the Niger Delta, similarly, it is suggested that it could have extended to the Isan oil field area.

5. Conclusion

The analysis of bitumen samples of Western Nigeria showed the presence of gammacerane which is a diagnostic marker for stratified water column a characteristic feature of the lake environment and has been inferred to be generated from Lower Cretaceous source rock. However, the samples of Isan-9 well also showed via transmitted white light microscopy the presence of botryococcus algae which is diagnostic for lacustrine precursor organic matter in the matrix of the source rock samples. In addition, the GC-MS analysis on extracts of the Isan-9 Well showed the presence of gammacerance in the m/z 191, which is diagnostic for stratified water column and a characteristic of a lake-type environment. The m/z 191 Mass Chromatogram also showed the presence of oleanane which is diagnostic for vascular plant materials. The presence of gammacerane and oleanane in the mass chromatogram is suggested to imply an ancient lacustrine lake system that was gradually modified as a result of sedimentary infill. It is possible that as the parts of the basin were transiting from lacustrine to marine it was accompanied by continental runoffs supplying vascular plant material to the basin.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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