

LECO Analytical Study of Subsurface Tertiary Shale Offshore Niger Delta

A.O. Aladesanmi,¹ A.Olowookere A.K Ogundana . J. Ochai

¹Department of Geology, Afe Babalola University, Ado Ekiti

Corresponding Author: A.O. Aladesanmi

Abstract: Leco C230 was used to determine the total organic carbon in dried and acidified subsurface tertiary shale samples in part of selected wells of offshore Niger delta. The samples is combusted at high temperature in an Oxygen rich atmosphere and converted all the carbon into carbon dioxide .The infrared detector was used to measure the mass of the carbon dioxide present and the mass is converted to percentage. The weight percentage of the carbon and sulphur in the samples were obtained. For carbon, the inorganic and organic carbon in weight percentage was summed up to give the total carbon content present in each sample. The ratio of sulphur to carbon content in each sample was obtained to provide interpretation of the depositional environment. The analysis shows that each sample has a higher carbon ratio content to sulphur in every sample analysed. The plot of sulphur carbon correlation was plotted. Sand and shale were the two lithologies present from the log interpretation, with shale occurring more. The presence of shale in the environment helped to decipher the environment as deep marine as shale is associated with high gamma count. Finally inference were made on the hydrocarbon potential of this samples with reference to past research work.

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I. Introduction

The Niger Delta Province contains only one identified petroleum system (Kulke, 1995; Ekweozor and Daukoru, 1994 ;). This system is referred to here as the Tertiary Niger Delta (Akata –Agbada) Petroleum System. The maximum extent of the petroleum system coincides with the boundaries of the province. The minimum extent of the system is defined by the areal extent of fields and contains known resources (cumulative production plus proved reserves) of 34.5 billion barrels of oil (BBO) and 93.8 trillion cubic feet of gas (TCFG) (14.9 billion barrels of oil equivalent, BBOE) (Petroconsultants, 1996a). Currently, most of this petroleum is in fields that are onshore or on the continental shelf in waters less than 200 meters deep, and occurs primarily in large, relatively simple structures. A few giant fields do occur in the delta, the largest contains just over 1.0 BBO (Petroconsultants, Inc., 1996a). Among the provinces ranked in the U.S. Geological Survey's World Energy Assessment (Klett and others, 1997), the Niger Delta province is the twelfth richest in petroleum resources, with 2.2% of the world's discovered oil and 1.4% of the world's discovered gas (Petroconsultants, Inc. 1996a). In 1908, the German Nigerian Bitumen Corporation drilled the first wells in the vicinity of the tar seep deposits in the northern portion of the delta (Frost, 1997). However, significant oil shows were not found in Tertiary rocks until the early 1950's. Shell-British Petroleum brought the first well on stream in 1958 at 5,100 barrels per day. From 1958 until the Biafran War in 1967, exploration and production increased in Nigeria. The war curtailed both activities until its end in 1970, when world oil prices were rising and Nigeria again could benefit economically from its petroleum resources in the Niger Delta. In 1971, Nigeria joined the Organization of the Petroleum Exporting Countries (OPEC) with a total production of 703 million barrels of oil (MMBO) per annum. In 1997, production rose to 810 MMBO (Energy Information Administration, 1998a). Thirty-one percent of this production (251 MMBO) was exported to the United States, making Nigeria the fifth largest supplier of U.S. oil. Despite the political uncertainty in Nigeria today, the country's sustainable production capacity is expected to increase over current production--they have agreed, however, to reduce their production by 225,000 barrels/day in 1998. Petroleum exploration is also expanding, especially in deeper water offshore, with the Nigerian government currently planning to offer six additional lease blocks in water up to 3000 m deep. Considering both oil and gas, the overall success ratio for exploration drilling is as high as 45% (Kulke, 1995;)Exploration of the small portions of the Niger Delta in Cameroon and Equatorial Guinea began much later than in Nigeria. Recoverable oil and gas (produced plus proved reserves) are much smaller than in Nigeria. In 1997, 96% of the Niger Delta recoverable petroleum was in Nigeria, 3.5% in Cameroon, and 0.5% in Equatorial Guinea (Petroconsultants, Inc., 1996a).

Location of the Area of Study

The Niger Delta is situated on the continental margin of the Gulf of Guinea in equatorial West Africa, between latitude 3° and 6°N and longitude 5° and 8°E, and extends throughout the Niger Delta Province as defined by Klett et al (1997). From the Eocene to the present, the delta has prograded southwestward, forming depobelts that represent the most active portion of the delta at each stage of its development (Doust and Omatsola, 1990). These depobelts form one of the largest regressive deltas in the world with an area of some 300,000 km² (Kulke, 1995), a sediment volume of 500,000 km³ (Hospers, 1965), and a sediment thickness of over 10 km in the basin depocenter (Kaplan et al, 1994). The well sample used for this work is located in the map on latitude 4° 20' and longitude 6° 02' .

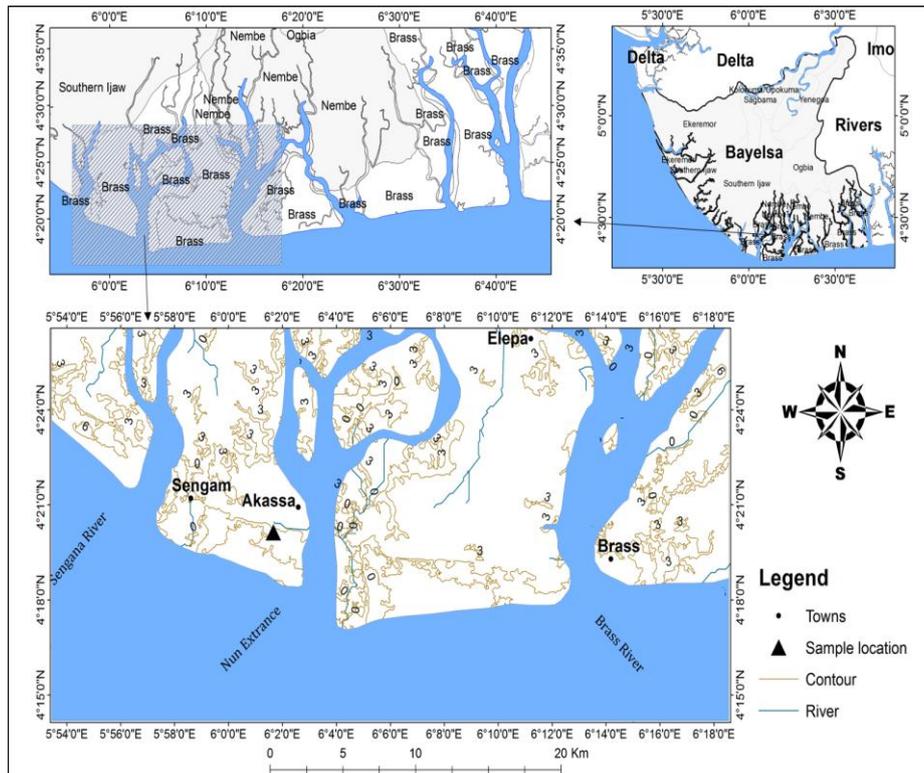


Figure 1: shows the Map of the study area created from the American Military Force Base Map.

Tectonic Settings

The tectonic framework of the continental margin along the West Coast of equatorial Africa is controlled by Cretaceous fracture zones expressed as trenches and ridges in the deep Atlantic. The fracture zone ridges subdivide the margin into individual basins, and, in Nigeria, form the boundary faults of the Cretaceous Benue-Abakaliki trough, which cuts far into the West African shield. The trough represents a failed arm of a rift triple junction associated with the opening of the South Atlantic. In this region, rifting started in the Late Jurassic and persisted into the Middle Cretaceous (Lehner and De Ruiter, 1977). In the region of the Niger Delta, rifting diminished altogether in the Late Cretaceous.

After rifting ceased, gravity tectonism became the primary deformational process. Shale mobility induced internal deformation and occurred in response to two processes (Kulke, 1995). First, shale diapirs formed from loading of poorly compacted, over-pressured, prodelta and delta-slope clays (Akata Fm.) by the higher density delta-front sands (Agbada Fm.). Second, slope instability occurred due to a lack of lateral, basinward, support for the under-compacted delta-slope clays (Akata Fm.). For any given depobelt, gravity tectonics were completed before deposition of the Benin Formation and are expressed in complex structures, including shale diapirs, roll-over anticlines, collapsed growth fault crests, back-to-back features, and steeply dipping, closely spaced flank faults (Evamy et al, 1978; Xiao and Suppe, 1992). These faults mostly offset different parts of the Agbada Formation and flatten into detachment planes near the top of the Akata Formation.

Lithology And Stratigraphy

The Cretaceous section has not been penetrated beneath the Niger Delta Basin, the youngest and southernmost sub-basin in the Benue-Abakaliki trough (Reijers et al, 1997). Lithologies of Cretaceous rocks deposited in what is now the Niger Delta basin can only be extrapolated from the exposed Cretaceous section in

the next basin to the northeast--the Anambra basin. From the Campanian through the Paleocene, the shoreline was concave into the Anambra basin (Hospers, 1965), resulting in convergent longshore drift cells that produced tide-dominated deltaic sedimentation during transgressions and river-dominated sedimentation during regressions (Reijers et. al, 1997). Shallow marine clastics were deposited farther offshore and, in the Anambra basin, are represented by the Albian-Cenomanian Asu River shale, enomanian-Santonian Eze-Uku and Awgushales, and Campanian/Maastrichtian Nkporo shale, among others (Nwachukwu, 1972; Reijers et. al, 1997). The distribution of Late Cretaceous shale beneath the Niger Delta is unknown. In the Paleocene, a major transgression (referred to as the Sokoto transgression by Reijers and others, 1997) began with the Imo shale being deposited in the Anambra Basin to the northeast and the Akata shale in the Niger Delta Basin area to the southwest. In the Eocene, the coastline shape became convexly curvilinear, the longshore drift cells switched to divergent, and sedimentation changed to being wave-dominated (Reijers et. al, 1997). At this time, deposition of paralic sediments began in the Niger Delta Basin proper and, as the sediments prograded south, the coastline became progressively more convex seaward. Today, delta sedimentation is still wave-dominated and longshore drift cells divergent (Burke, 1972). The Tertiary section of the Niger Delta is divided into three formations, representing prograding depositional facies that are distinguished mostly on the basis of sand-shale ratios. The type sections of these formations are described in Short and Stäuble (1967) and summarized in a variety of papers (e.g. Avbovbo, 1978; Doust and Omatola, 1990; Kulke, 1995). The Akata Formation at the base of the delta is of marine origin and is composed of thick shale sequences (potential source rock), turbidite sand (potential reservoirs in deep water), and minor amounts of clay and silt. Beginning in the Paleocene and through the Recent, the Akata Formation formed during lowstands when terrestrial organic matter and clays were transported to deep water areas characterized by low energy conditions and oxygen deficiency (Stacher, 1995). Little of the formation has been drilled; therefore, only a structural map of the top of the formation is available. It is estimated that the formation is up to 7,000 meters thick (Doust and Omatsola, 1990). The formation underlies the entire delta, and is typically overpressured. Turbidity currents likely deposited deep sea fan sands within the upper Akata Formation during development of the delta (Burke, 1972). Deposition of the overlying Agbada Formation, the major petroleum-bearing unit, began in the Eocene and continues into the Recent. The formation consists of paralic siliciclastics over 3700 meters thick and represents the actual deltaic portion of the sequence. The clastics accumulated in delta-front, delta-topset, and fluvio-deltaic environments. In the lower Agbada Formation, shale and sandstone beds were deposited in equal proportions, however, the upper portion is mostly sand with only minor shale interbeds. The Agbada Formation is overlain by the third formation, the Benin Formation, a continental latest Eocene to Recent deposit of alluvial and upper coastal plain sands that are up to 2000 m thick (Avbovbo, 1978)

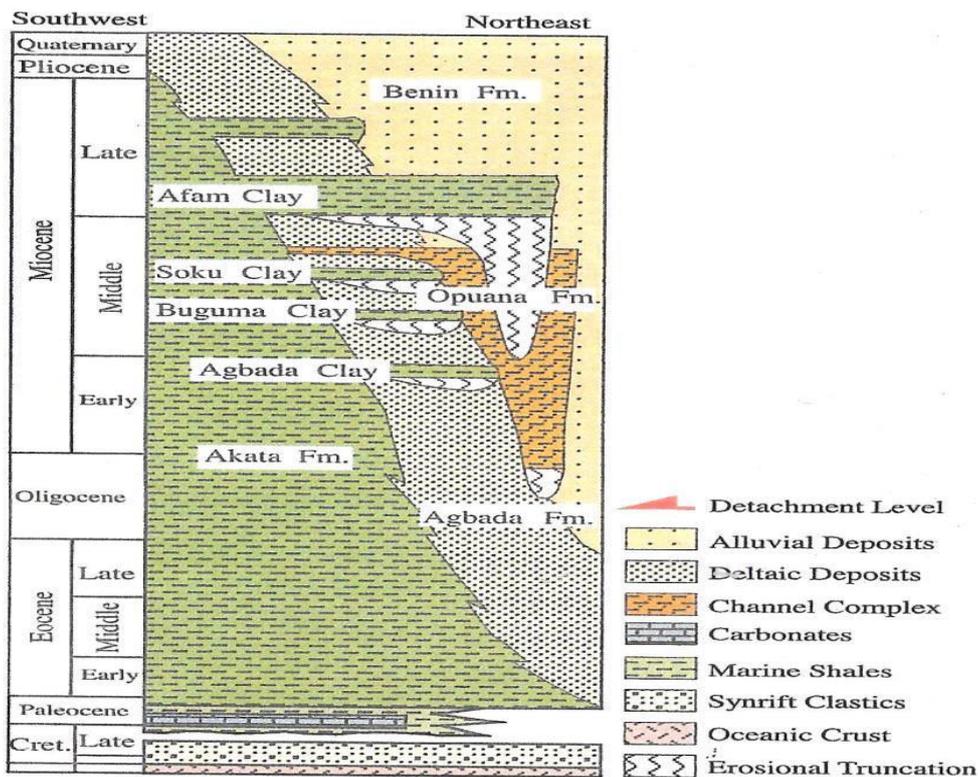


Figure 2: (Stratigraphy representation of Niger delta)

II. Material And Method

Data Availability

The data acquisition for this research comprises conventional well log and Base map of the Area. This were collected and validated from relevant authorities. Base Map contains reference information of how to locate and assess the study area. It has a scale, geographic co-ordinate, which helps us to locate the two major wells involved in the course of this research, the co-ordinates of this subsurface wells are N 4° 20' and E 6° 8'. Well log data suite comprises of data sheet available for the evaluation of the wells.

From these data, good estimates may be made of probable hydrocarbon bearing zones or suspected hydrocarbon reservoirs. They are Lithology log, Resistivity log and Porosity log.

Sample Collection and Laboratory Analysis

The Ditch cuttings samples were collected at two major oil wells in the areas from Adaxx Petroleum Nigeria Limited. At each point of collection, co-ordinates were taken and the samples were packed in polythene bags and labelled. The samples were kept in sample bags to minimize contamination and labelled to avoid mix up of samples. The laboratory analyses carried out are of Three aspects namely: Pulverization of samples (per 100g), Carbon and Sulphur by LECO and Organic/Inorganic Carbon.



Figure3:LECO CS230 Analyzer for Total Organic Carbon

Procedure and Experiment

To run LECO analysis the Carbon detected must be in form of combustion and infrared detection. All glassware and ceramic ware used in sample processing are combusted at 400°C for at least 4 hours. Rock samples (ditch cuttings) are obtained within a range of 20-25 or 30 grams and remain frozen at -20°C until processing. The samples are crushed into fine powder in a tempered chrome steel, mortar grinder machine for ten seconds. The sample powder is carefully removed with the aid of a brush, brushing them out into sample containers already labelled. Homogenize the sampler (i.e mixing it) if the grain has not yet been crushed properly into powder forms. An aliquot of dried, homogenized sample is placed in an aluminum-weighing pan and dried at 105°C. Thereafter the sample powder can be split mechanically using granulometry analysis to ensure that the grain size is < 63µm or 250 mesh. Finally, 2grams of the powdered sample is collected for 3×10 second grinding to prepare for infrared spectrophotometry analysis and packed aside. The spectrometer subjects the samples to 3×10 for each grinding to look at the clay mineralogy. The other remaining powder sample is grinded 2×10 second and stored in a glass bottle for other geochemical analysis. The LECO CS230 Carbon Analyzer is calibrated prior to the analysis of samples. Different amounts of high purity calcium carbonate standard (99.95% purity, carbon content of 12.0%) are used to calibrate the instrument. The approximate amounts of calcium carbonate used for the six-point calibration are; 0.01 g, 0.05 g, 0.10 g, 0.25 g and 0.50 g. An empty carbon-free combustion boat is analyzed as a blank for the calibration curve. The calibration curve provides an analysis range of approximately 0.0 to 0.06 g

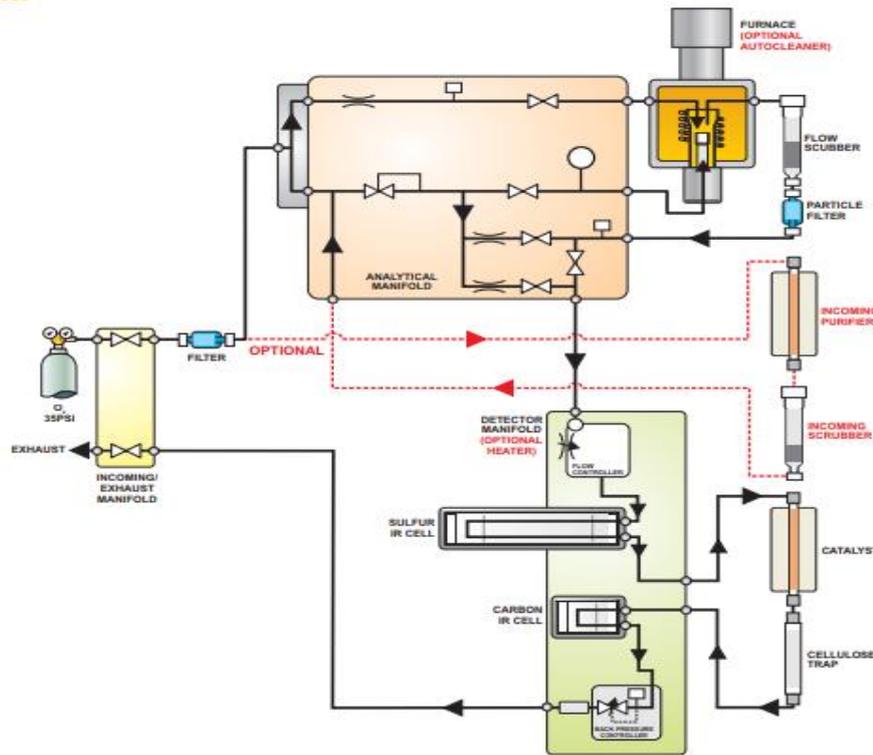


Figure 4:Flow diagram showing Carbon/Sulfur Analysis process from LECO Corporation

Experimental Results and Discussion

The Parameters determined using LECO CS230 analyzer are; Total Carbon (TC%) Total Sulphur (TS %Total), Inorganic Carbon content (IC %) and Total Organic Carbon (TOC%)

Sample depth(m)	Parameter (value in %)			
	S(Total)	C(Total)	TOC	IC
10720-10750m	1.63	5.39	2.53	2.86
11210-11240m	2.27	7.83	3.01	4.82
11660-11690m	3.5	4.88	3.15	1.73
13260-13280m	2.49	6.01	1.63	4.38
13598-B	2.73	6.23	2.37	3.86

Table 1: Summary of Leco analyser results obtained from different samples

Organic Richness (TOC)

The result of the five shale samples shows an appreciable amount of carbon richness. The organic richness represented by the total organic carbon (TOC) ranges from 1.63 to 3.15 wt %). The studied sample falls under the range of ‘good to very good’ in its organic richness (from the standard range determination for organic richness). The average mean of the total carbon content is 6.07wt%. The percentage weight of the Organic carbon content increases with an increase in depth from sample 1 to 3 until an abrupt change in sample 4 where it reduces in percentage and then peaks up again in sample 5. The Organic richness are controlled by concentration and nature of oxidizing agents, type of organic matter deposited and the sediment-accumulation rate (Rondeel 2002). The table below shows a range of values used in the determination of organic richness level present in each sample.

Organic Richness	TOC Value(%Weight)
Poor	0-0.5
Moderate	0.5-1.0
Good	1.0-2.0
Very Good	2.0-5.0
Excellent	5+

Table 2: showing Standard range values of the Organic richness

Sulphur isotopic composition (S total)

Sulphur isotopic composition of the sample range from 1.63wt% in sample 1 to 3.5wt% in sample 3. The average mean of the sulphur contents of the well is 2.5wt%. Also as explained for carbon compositions of each samples, the sulphur content increases from sample 1 to sample 3 and then reduces in sample 4 and finally increases again in the last sample but the change in the composition of sulphur was not at equilibrium with a change in the carbon content as seen that they both occurred at different depths and of course analysed for different samples. High sulphur percentage could be associated with marine deposition, because fresh waters are usually low in sulphate and sulphur is derived mainly from sulphate that was reduced by anaerobic bacteria in the absence of Iron (Fe +2) ions.

Sulphur Carbon Ratio (S:C)

The ratio of sulphur to carbon can be obtained for each of the samples analysed. A graph showing the total sulphur content to the total carbon content present in each sample plotted showed the relationship between the two elemental compositions present in the individual samples. The ratios of sulphur to carbon in each of the samples are shown in the table below.

S/N	Sample Depth(ft)	S:C
1	10720-10750	02:05
2	11210-11240	01:04
3	11660-11690	01:03
4	13260-13280	03:05
5	13598-?	01:03

Table 3: Showing Sulphur and Carbon Ratio with increase depth

It is seen from the table above that in every sample an increase in the isotopic composition of sulphur present in the sample, there is about thrice an increase in the isotopic composition of carbon. This could suggest that the environment where these samples were obtained is rich with higher percentages of carbon compared to the sulphur content present. There was a change in the trend at the 13260ft in sample 4 where the percentage weight of the sulphur shoot up, this could be associated with the reduction process of sulphate by anaerobic bacteria in the absence of Iron (II) ions in the deep marine environment. Thus if this sample were to be tested for their hydrocarbon potentials, it could be possible that they could contain hydrocarbon due to its organic carbon richness. This also satisfies past research works which claim the presence of very little sulphur content in Nigeria’s oil but higher content in the organic carbon richness (Niger delta has organic richness between 3-7 which means it is very rich in its organic content and would be classified in the range of good to very good using the standard organic richness scale).

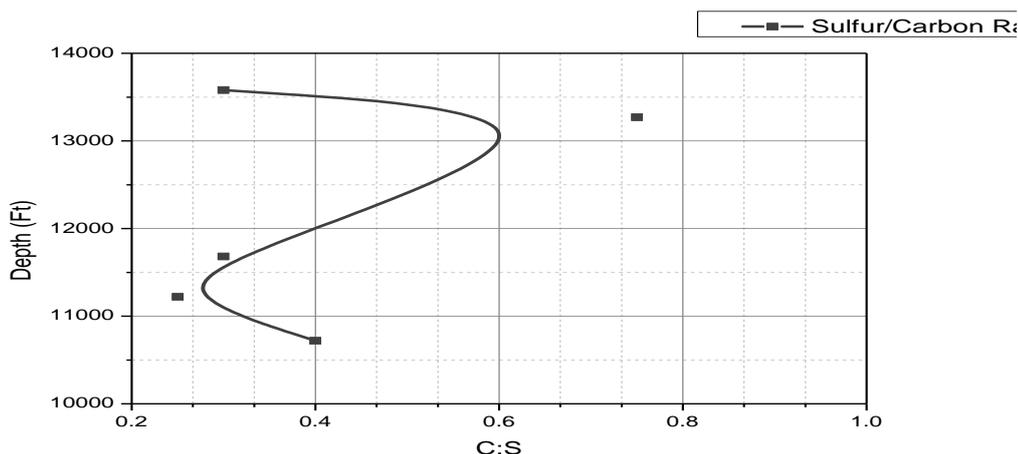


Figure 5: Graph Plot For Sulphur Carbon Ratio with increase depth

III. Conclusion and Recommendation

The geochemical studies of the Total carbon (organic and inorganic) indicate deposition of these shales under a Marine environment with some terrestrial input. The studied shale’s have good to very good organic richness most likely type III/II kerogen (common with Niger delta). They indicate the capability to produce gas and fair oil source. The TOC content of the shale averages between 3.15 to 1.63 wt% and suggest possible oil even with its relatively low concentration of sulphur. Taken together with the result from other studies, it was

concluded that the shale formed in the deep marine environment and process of anaerobic bacteria caused the amount of sulphur under anoxic condition by deeply buried down dipping marine/ lacustrine source rocks most likely from a hypersaline paleoenvironment. Judging from the chemistry of the shale, it is very possible to say that its high richness of organic carbon could mean a possibility in hydrocarbon richness.

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