International Journal of
Science Inventions Today
www.ijsit.com
ISSN 2319-5436
Research Article

ORGANIC MATTER QUANTITY, QUALITY AND THERMAL MATURATION OF CRETACEOUS SOURCE ROCKS IN PARTS OF AFIKPO BASIN, SOUTHEASTERN, NIGERIA

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ABSTRACT

The organic geochemical evaluation of Cretaceous shale comprising Nsukka and Mamu Formation which cropped out in parts of Afikpo Basin was evaluated for organic matter quantity, quality and thermal maturation. Total Organic Carbon (TOC), Soluble Organic Matter (SOM), free hydrocarbon (S1) and pyrolysis temperature (Tmax) were assessed using Rock-Eval pyrolysis and magnetic agitation methods. The results showed that the TOC values varied from 0.58-0.69wt% for samples analyzed from Obotme area, 0.60-0.61wt% for Nkana area, 0.67-0.71wt% for Okobo area, 0.68-0.78wt% and 7.36wt% for Amuvi and Ututu areas respectively. The average TOC value is 1.05wt% for all samples. SOM values varied from 100ppm to 375ppm with an average value of 203ppm. In the study area the results also showed that samples analyzed from Obotme area has Tmax values ranging from 442^{0}C-464^{0}C, indicating early to peak maturity. Tmax values of samples analyzed from Amuvi, Nkana and Ututu areas varied from 364^{0}C-429^{0}C, indicating thermal immaturity, samples analyzed from Okobo area indicate early thermal maturity with Tmax values ranging from 427^{0}C-448^{0}C.The average Tmax value for all sample is 414^{0}C. The Tmax values in the study area shows that the sediments are immature and have not reached the oil generation phase, but are within the gas phase. The organic matter quantity is adequate for sediments to yield hydrocarbon whereas the organic matter quality is inadequate to expel hydrocarbon.

INTRODUCTION

Location of Study area: The study area is located in Afikpo Basin, Southeastern part of Nigeria (Figure 1.1). It stretches from longitude 07^{0} 54^{1} 23.3^{11}E to 07^{0} 49^{1} 90.2^{11}E and latitude 05^{0} 22^{1} 40.1^{11}N to 05^{0} 27^{1} 81.5^{11}N. The areas covered include Nkana and Obotme in Akwa Ibom State and Amuvi, Okobo, and Ututu in Arochukwu LGA of Abia State (Figure 1).

AIM AND OBJECTIVES OF STUDY

The aim of this research work is to carry out an organic geochemical investigation on shale samples collected from the study area.

The objectives of the study include the following.

1. To determine the quantity of organic matter in the shale samples.

2. Assess the thermal maturation of the shale sample

3. Evaluate the quality of the organic matter in the samples.

4. Determine the hydrocarbon source potential of the shale samples

STRATIGRAPHIC EVOLUTION:

Murat, (1972) described the stratigraphy of the Afikpo Basin and noted that the sedimentation was controlled dominantly by transgressions and regressions episodes. The Afikpo Basin in the Southern Benue Trough has the following lithostratigraphic divisions;

(a) Asu River group: The Asu River Group consists of shales, sandstones and limestone. It is the oldest lithostratigraphic unit in the study area, and was deposited during the Albian transgressive phase. It is also the oldest dated sedimentary rock unit in Southern Benue Trough. According to Reyment (1965), the Albian sediments were moderately folded in many places with the fold axis trending NE-SW. Ukaegbu and Akpabio (2009) have differentiated the Asu River Group northeast of the Afikpo Basin. According to them, the Asu River Group consists of alternating shale, siltstone with occurrence of sandstone. The maximum thickness of Asu River Group is 1000m, Albian in age and rich in ammonites as well as foraminifera, radiolarian and pollens. The shale, Asu River Group is characterized by species of *Monticeras* and *Elobiceras* which are ammonites (Offodile, 1976).

(b) Ezeaku Formation: The typical locality of the Eze Aku Group is the Eze Aku River valley in southeast of Eze Aku. Murat (1972) was of the view that the Eze-Aku shale shows deposits of marine conditions in a tectonically controlled Basin. He believed that sandstone deposits mark a period of regression, while the shale deposits indicate a period of transgression.

(c) Agwu Shale: The Agwu shale overlies the Eze-Aku Formation conformably. The lithology is a blurish-grey well bedded shale interbeded with fine grained yellow calcareous sandstone and shaly limestones, with a total thickness of about 900meters.

(d) Nkporo Shale: In Southeastern Nigeria, Campanian sediments probably belong to the base of the Nkporo Formation. No typical Campanian Ammonites have been found in Nigeria but it is probably that the base of the Nkporo Formation and its lateral equivalents, are Campanian in age (Ukaegbu *et al*., 2009). The major part of the Formation is Meastrichtian. The Campano-Maastrichtian sediments in Southeastern Nigeria have dark grey shale, which is often friable. This part of the sequence belongs to the Nkporo Formation. The Owerri sandstone and Enugu shale are lateral equivalents of the Nkporo Formation.

(e) Mamu Formation: It is stratigraphically synonymous to the lower coal measures and is of Lower-Middle Maastrichtian in age. Cross stratification is rare and of very low angle when present. There are fragment interbeds of carbonaceous shales with a sparse arenaceous micro fauna and coal beds. The lithology of Mamu Formation in the subsurface is similar to that on the surface except that the more continuous and thicker subsurface sections show that there are numerous fairly thick shale interbed in the sequence. .

LITERATURE REVIEW

PREVIOUS WORK:

Many studies have documented the methodology of geochemical evaluation of some rocks. Geochemical studies by Omoboriowo *et al* (2012), revealed that the organic carbon values of samples taken from the Arochukwu area are greater than the threshold value. They revealed that shale samples from the lower Benue Trough exhibits characteristics of potential hydrocarbon source rocks, and noted series of oil and gas shows found within some of the beds. According to Petters *et al* (1982), the black shale of the Benue Trough has high terrestrial organic matter. The TOC values range from 0.5 to 7.4wt%. Whereas the minimum value of TOC for potential source rocks is 0.5wt% (Tissot *et al*., 1978). Based on this minimum value of TOC for potential source rocks, the shales under study have low to high generation potential. The high SOM (Bitumen) and saturated hydrocarbon extracted from the shale supports the high potential source rock of the study area. They concluded that some of the shale samples in the lower Benue Trough has features of good source rock properties and may have generated oil before upliftment to their present shallow stratigraphic position as a result of the Santonian tectonism.

Various studies by Van krevelene (1961) and Tissot *et al* (1974) confirm that the chemical composition of finely disseminated organic material originally in a marine environment shows a tendency to be transformed into hydrocarbon easily and efficiently than woody, carbonaceous and terrestrial materials.

Lignite kerogen is generally considered to be a poor source and hydrocarbons produced are mainly dry gas type while sapropelic kerogens, under thermal influence are more likely to yield oil and wet gas than dry gas through low to moderate levels of diagenesis. With increased diagenesis (metagenesis) only dry gas may be produced.

Lambert *et al* (1984), in their paper on the Petroleum Source bed Evolution of the Niger Delta, used organic matter content, maturity and vitrinite reflectance successfully to examine and suggest the possible source rocks in the Niger Delta.

MATERIALS AND METHODS

(A) FIELD STUDY:

This involved outcrop observation, description and collection of rock samples for laboratory analysis. A survey of the site was done on the first day of the field work, the field work spanned five days.

Sample collection: The spot sampling (Davies *et al*, 1973) method was adopted for data collection during the field work. 17 samples were collected using steel hand-auger at different depths for vertical delineation of change in organic matter content as observed visually. All samples were put inside cellophane bags and labeled properly to avoid mix-up.

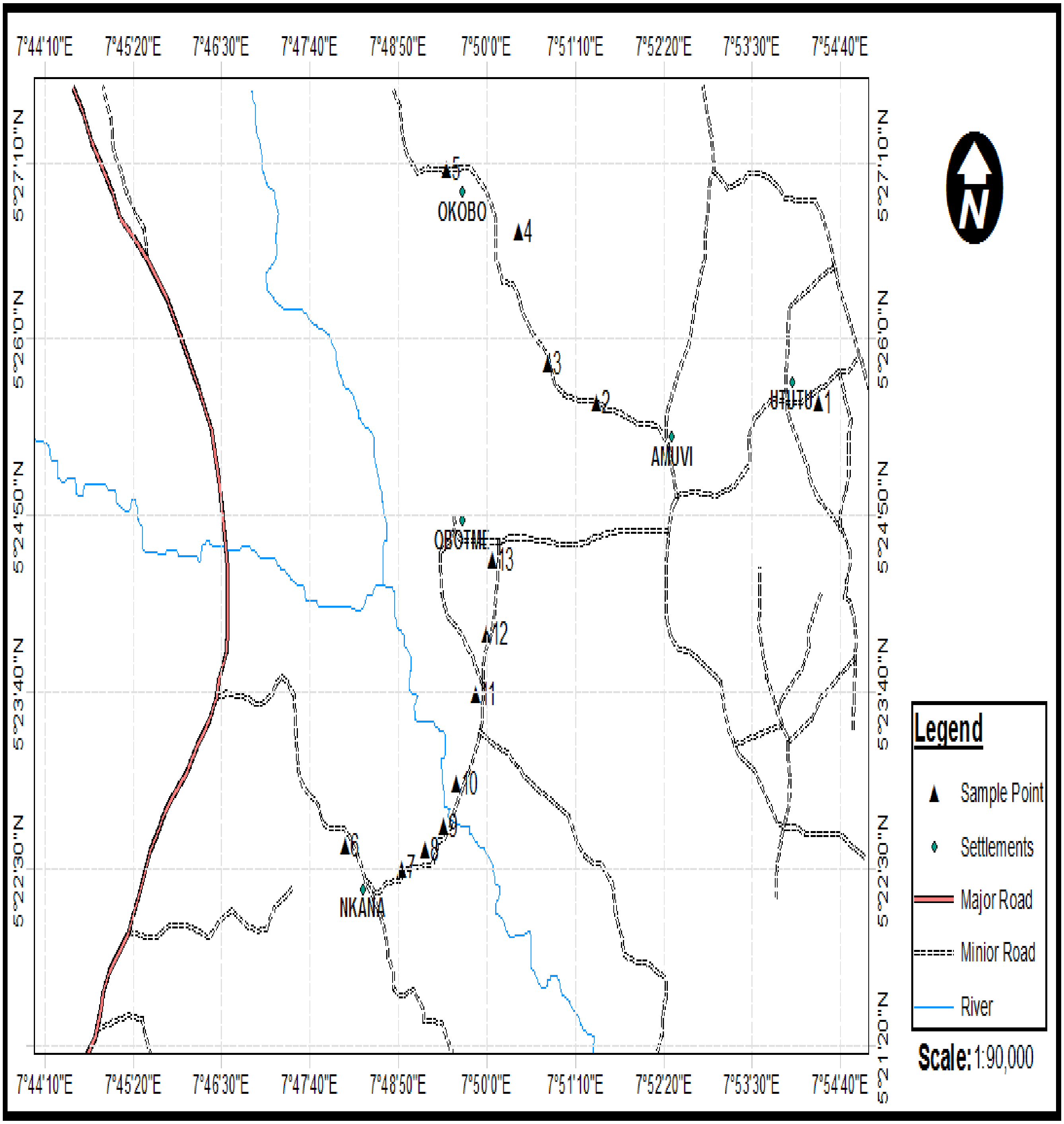

Figure 1: Map showing sampling points in the study area

(B) LABORATORY METHODS:

Standard laboratory methods as applied in petroleum potential studies were adopted. This involved analysis for Total Organic Carbon (TOC), Soluble Organic Carbon (SOM), Pyrolysis temperature (Tmax) and

Free hydrocarbon (S1).The number of samples evaluated are seven

Soluble organic matter (SOM): The analysis for Soluble Organic Matter (SOM) was carried out in the Plant Anatomy and Physiology Research Laboratory, University of Port Harcourt. This was to determine the petroleum potential of the shales which make up the Nsukka and Mamu Formations. The methodology used for acquiring data for the SOM is the Magnetic Agitation Method (magnetic extraction). The reagents and equipment used include chloroform, conical flask, meter balance, spatula, graduated cylinder, boiling tube, aluminium foil, filter paper, spectro-photometer, magnetic stirrer and funnel.

Rocks-eval pyrolysis/Toc analysis: Rock-Eval pyrolysis technique was applied in this work and is based on the methodology described by Espitalié *et al* (1977). This was carried out in the Civil Engineering and Geosciences Laboratory, Newcastle University. This technique provided data on the quantity, quality, and thermal maturity of the associated organic matter.

Procedure:

1. Each of the 17 pulverized samples were one at a time weighed (100.0 mg) into stainless steel crucibles (SPI-Glass Crucibles).

2. Each of the crucibles was placed in the programmed Delsi 5 pyrolysis unit.

3. The samples were heated under an inert atmosphere of helium at 300º C for 3-4 minutes.

4. After which there was pyrolysis of the samples at 25ºC/minute to 600º C, followed by posterior cooling down for the next sample to be run (Figure 3.3). Figure 3.3 is an example of a pyrogram during the process of sample pyrolysis.

5. The generated data was sent to a connected computer set.

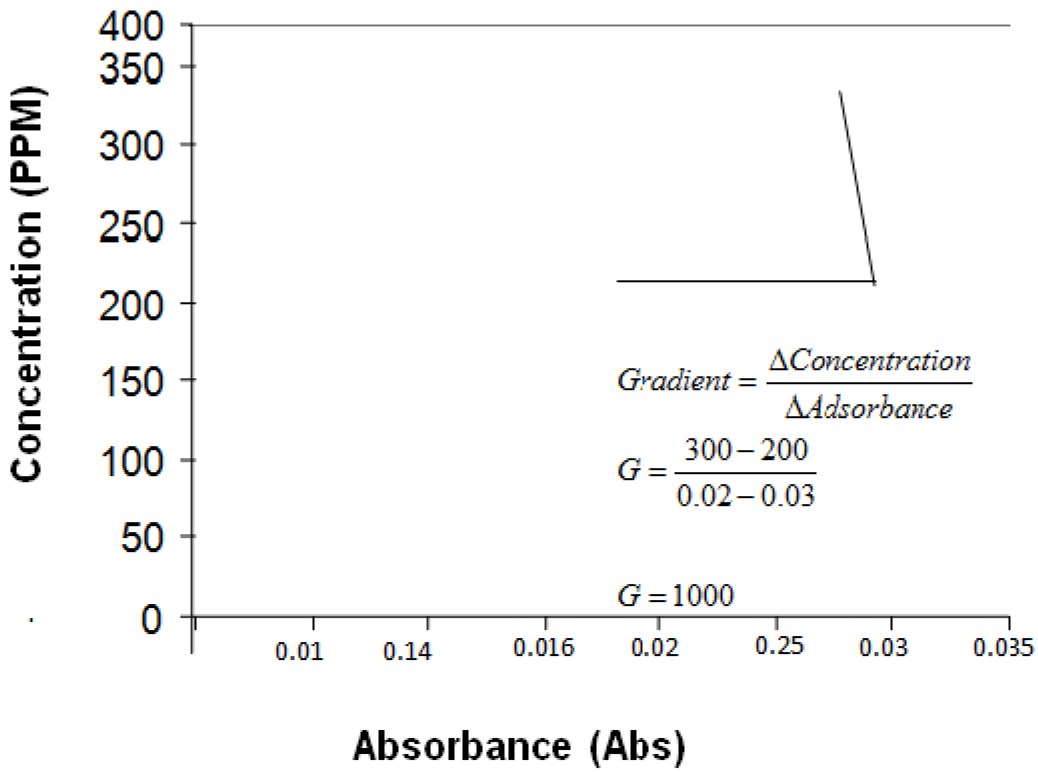

Figure 2: Graph of concentration versus absorbance

RESULTS AND DISCUSSION

FIELD WORK RESULTS:

The results of fieldwork are presented below.

Lithologic Description:

The lithologic description of location 1 to 13 from the fieldwork is presented below.

4.2.1 Location 1	**Ututu.**
Depth (m)	Lithologic description (Figure 1)
1	Top soil; consist of medium grained sand
2.5	Fine to medium grained ferruginous sandstone. They are reddish.
7	Medium grained sand with clasts. They are whitish.
15	Well bedded clay. They are brownish.
20	Shale, dark to gray.
4.2.2 Location 2	**Amuvi.**
Depth (m)	Lithologic description.
0.5	Top soil; consist of medium grained sand.
0.7	Shale, dark gray.
1	Limestone; whitish with brownish to yellow patches.
4.2.3 Location 3	**Amuvi.**
Depth(m)	Lithologic description.
0.5	Top soil; consist of weathered materials, brownish.
1.0	shale; dark gray.
1.5	Limestone; whitish with brownish to yellow patches

Figure   Summary of lithological logs of location 1,6 and 7 (Ututu-Nkana area).

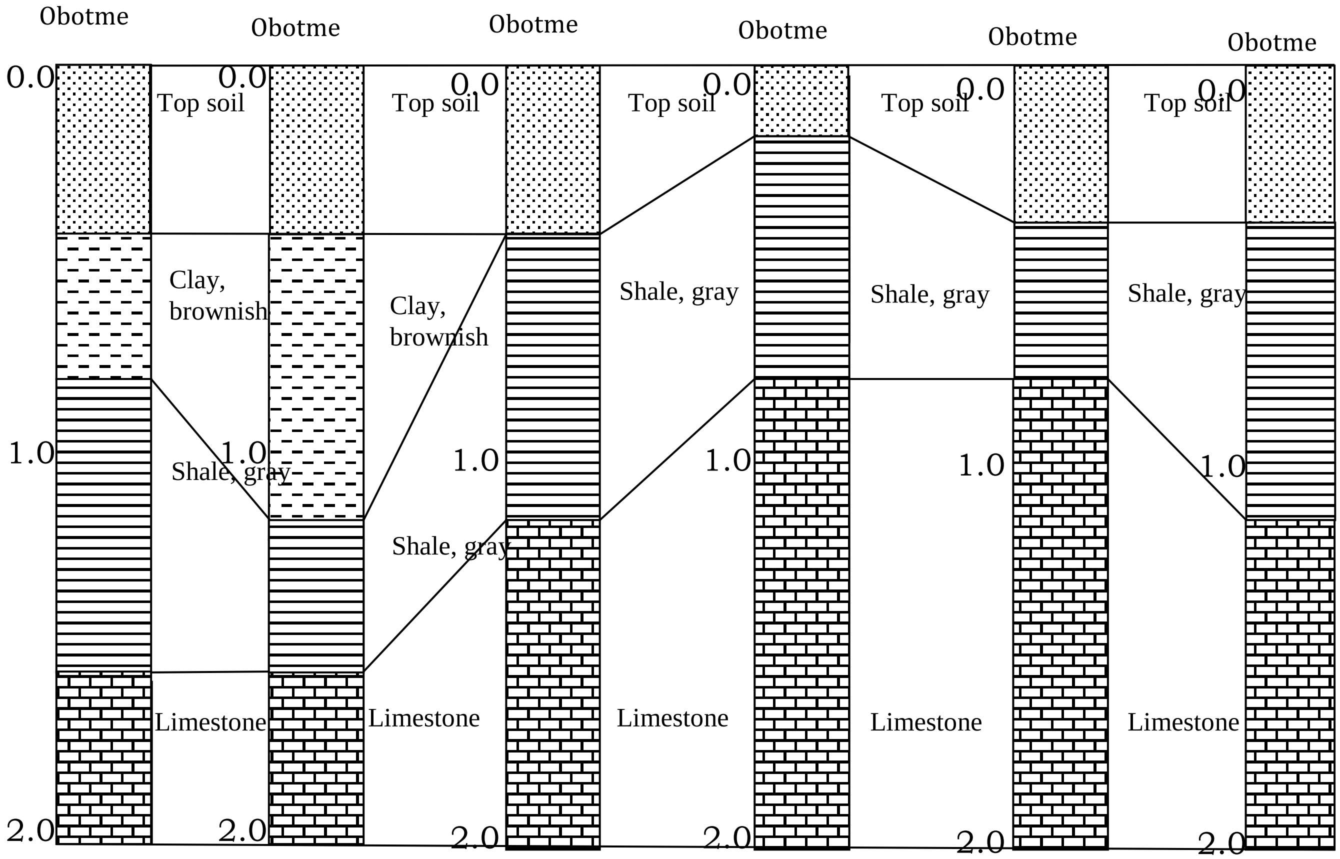

Figure 3: Summary of lithological logs of location 8-13 (Obotme area)

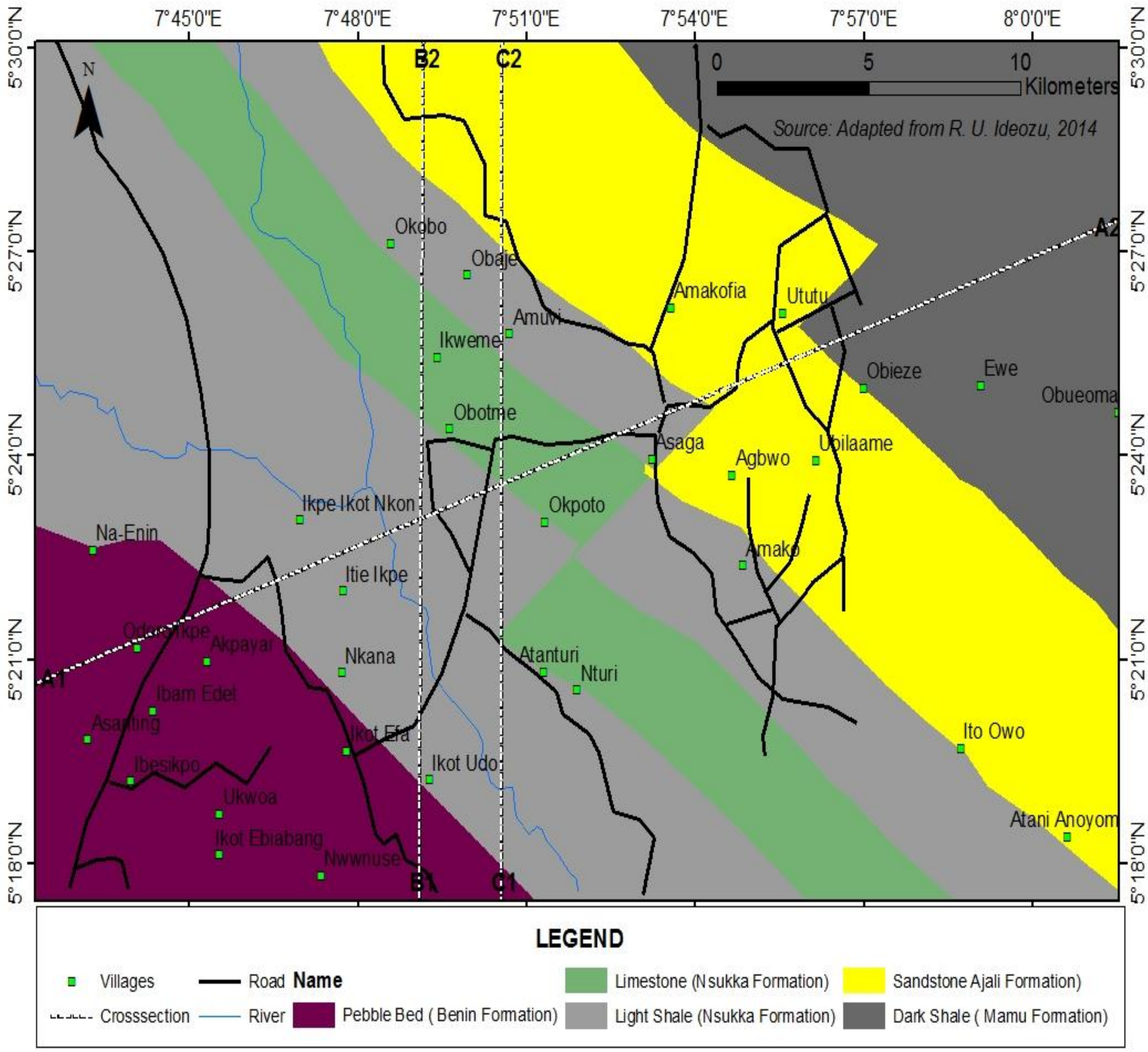

Figure 4: Geologic map of the study area (Modified after Ideozu, 2014)

LABORATORY ANALYSIS RESULTS:

Rock-Eval pyrolysis result

SAMPLE No	Lithology	Location	Formations	Depth (m)	TOC (wt%)
1	Shale	Ututu	Mamu	13	7.36
2	Shale	Amuvi	Nsukka	0.2	0.68
3	Shale	Amuvi	Nsukka	1.0	0.78
4	Shale	Okobo	Nsukka	1.0	0.67
5	Shale	Okobo	Nsukka	5.5	0.71
6	Shale	Nkana	Mamu	4.5	0.62
7	Shale	Nkana	Mamu	4.0	0.60
8	Shale	Obotme	Nsukka	1.0	0.61
9	Shale	Obotme	Nsukka	1.5	0.62
10	Shale	Obotme	Nsukka	0.5	0.58
11	Shale	Obotme	Nsukka	1.0	0.62
12	Shale	Obotme	Nsukka	0.5	0.65
13	Shale	Obotme	Nsukka	1.0	0.67
14	Shale	Obotme	Nsukka	2.0	0.58
15	Shale	Obotme	Nsukka	2.0	0.88
16	Shale	Obotme	Nsukka	0.5	0.58
17	Shale	Obotme	Nsukka	1.0	0.69

Table 1: Total Organic Carbon (TOC) results.

Depth (m)	Lithology	Location	Formation	SOM (ppm)	TOC (wt%)	$S_{1(mg/g)}$	Tmax
13	Shale	Ututu	Mamu	300	7.36	0.21	424
0.2	Shale	Amuvi	Nsukka	150	0.68	0.20	423
1.0	Shale	Amuvi	Nsukka	160	0.78	0.04	429
1.0	Shale	Okobo	Nsukka	375	0.67	0.03	448
5.5	Shale	Okobo	Nsukka	375	0.71	0.04	427
4.5	Shale	Nkana	Mamu	300	0.62	0.04	364
4.0	Shale	Nkana	Mamu	375	0.60	0.03	364
1.0	Shale	Obotme	Nsukka	150	0.61	0.05	354
1.5	Shale	Obotme	Nsukka	120	0.62	0.09	424
0.5	Shale	Obotme	Nsukka	150	0.58	0.03	460
1.0	Shale	Obotme	Nsukka	200	0.62	0.01	442
0.5	Shale	Obotme	Nsukka	150	0.65	0.02	456
1.0	Shale	Obotme	Nsukka	180	0.67	0.03	464
2.0	Shale	Obotme	Nsukka	175	0.58	0.02	372
2.0	Shale	Obotme	Nsukka	175	0.88	0.02	366
0.5	Shale	Obotme	Nsukka	120	0.58	0.09	405
1.0	Shale	Obotme	Nsukka	100	0.69	0.03	410

Legend

Yellow	Immature
Blue	Early Mature to peak mature
Green	Late mature

Table 2: Summary of Rock-Eval/TOC and SOM results

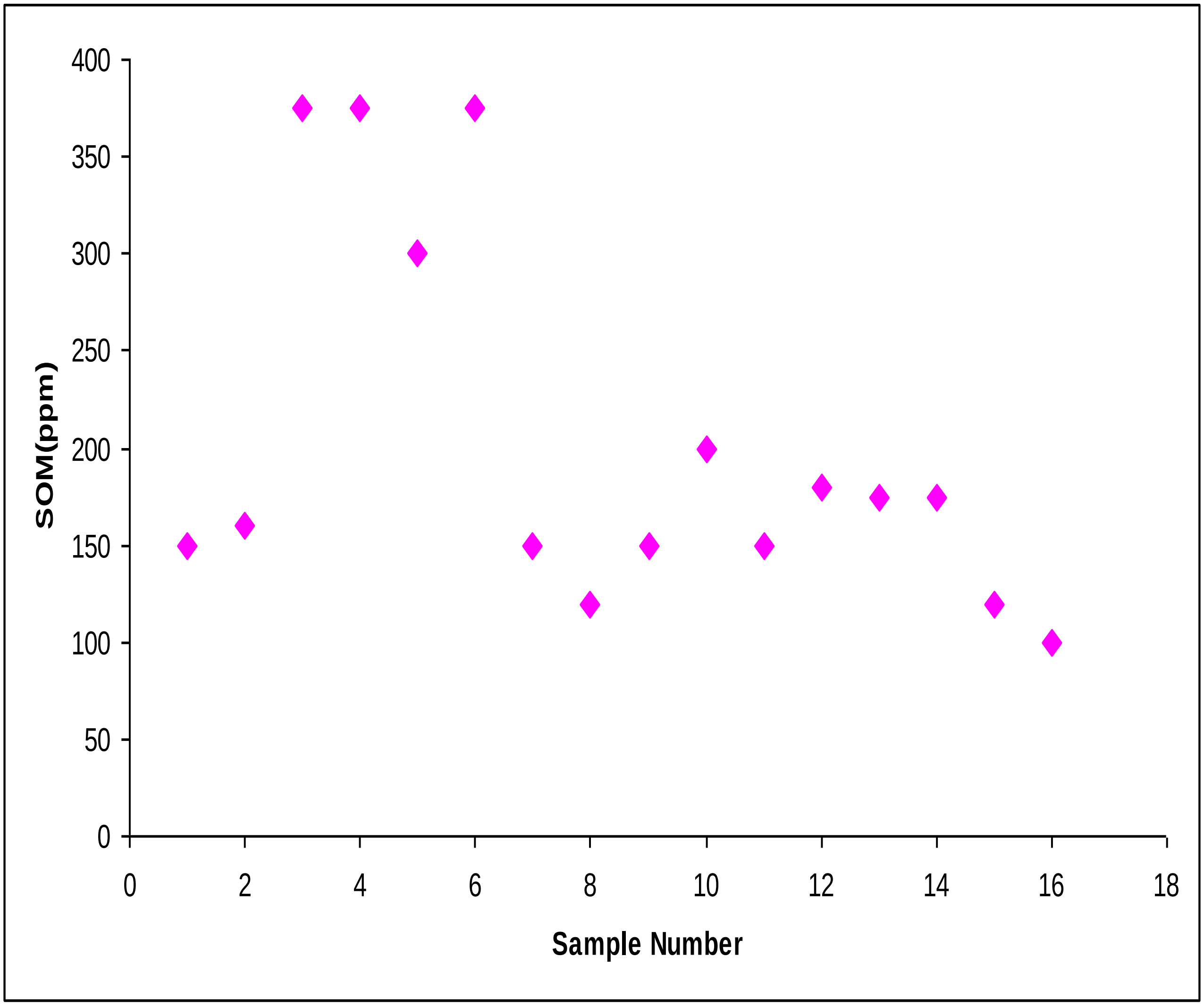

Figure 5: Varriation of SOM (ppm) with sample intervals (after Dow, 1977)

(a) Total organic carbon (TOC): The Total Organic Carbon (TOC) is a direct measure of the organic matter present in the rock. It is indicative of the organic matter available for the formation of hydrocarbon. The results of the TOC are present in Table . The TOC values varied from 0.58-0.69wt% for samples collected from Obotme area, 0.60-0.61wt% for samples collected from Nkana area, 0.67-0.71wt% for samples collected from Okobo area, 0.68-0.78wt% and 7.36wt% for samples collected from Amuvi and Ututu areas respectively. The Nsukka Formation was traced from Obotme area to Okobo area while the Mamu Formation include Nkana and Ututu areas. The Nsukka Formation has TOC value ranging from 0.58-0.88wt% with average value of 0.66wt%, while the Mamu Formation has TOC value ranging from 0.60-7.36wt% with average value of 2.86wt%. The average TOC value is 1.05wt% for all the samples analyzed. The results show that the samples have TOC greater than 0.5wt% which is the minimum threshold value for hydrocarbon generation in siliclastic rocks (Tissot *et al*, 1978). These values are indicative of good organic matter richness for hydrocarbon generation. The results suggest that only one sample collected from Ututu area is capable of expelling hydrocarbon even at minimal maturity.

(b) Soluble organic matter: Seventeen shale samples yielded organic extract in the range of 100ppm-375ppm with an average value of 209ppm for all the samples analyzed. The results showed that the SOM value varied from 100-200ppm for samples collected from Obotme area, 300-375ppm for samples collected from Nkana area, 375ppm for samples collected from Okobo area, 150-160ppm and 300ppm for samples collected from Amuvi and Ututu sections respectively. The Nsukka Formation has an SOM range of 100-375ppm with an average value of 184ppm, while the Mamu Formation has SOM range of 300-375ppm with an average value of 325ppm. The work of Deroo *et al* (1977), relates the SOM contents to source rock potential. These values are greater than 50ppm and it indicates that the source rocks have adequate organic matter to generate hydrocarbon.

(c) Residual hydrocarbon (S1): The S1 values (see tabe4.4) varied from 0.01-0.09mg/g for samples collected from Obotme area, 0.03-0.04mg/g for samples collected from Nkana area, 0.03-0.04mg/g for samples collected from Okobo area, 0.02-0.04mg/g and 0.02mg/g for samples collected from Amuvi and Ututu areas respectively. The Nsukka Formation has an S1 range of 0.01-0.09mg/g with an average value of 0.04mg/g, while the Mamu Formation has S1 range of 0.02-0.04mg/g with an average value of 0.03mg/g. The results shows that only little amount of hydrocarbon might have been generated by the rocks in the subsurface.

(d) Pyrolysis temperature (Tmax): In this study, Tmax value is used as a simple measure of the samples' level of thermal maturity. Thermally matured samples in the study area displayed Tmax value of at least 442^{0}C, with an average value of 414^{0}C for all the samples analyzed. This is in line with minimum maturity threshold of 435^{0}C, Peters *et al* (1986). Tmax values range between 442-464^{0}C for samples collected from Obotme area indicating early to peak maturity. Tmax values for samples collected from Amuvi area varied from 423- 429^{0}C indicating thermal immaturity. Samples collected from Okobo area varied from 427- 448^{0}C indicating immature to early thermal maturity. Samples collected from Ututu area showed Tmax value of 364^{0}C indicating thermal immaturity. Thermal maturation level appeared to be more elevated in the samples from Nsukka Formation, thus Nsukka Formation in the study area is more mature as opposed to Mamu Formation which is older and buried deeper. This maturity anomalous between the two Formations may have resulted from local hot spot associated with tectonism in the study area.

Based on the results of geochemical analysis, the organic matter content of the samples is adequate for hydrocarbon generation. The samples analyzed from Obotme, Nkana, Okobo and Amuvi are classified as poor, while the sample analyzed from Ututu area is classified as very good. There is contrasting levels of thermal maturity in the study area. Maturity variation in the study area is due to presence of high temperature associated with tectonism. The results indicate an oil and gas source possibility in Nsukka Formation, while Mamu Formation in the study area has potential for gas only.

CONCLUSION

Based on the data generated from field and laboratory analysis, the following conclusions are reached.

1. The Ututu area is thermally immature, and contains TOC value of 7.36wt%.This suggests that this area has poor hydrocarbon source potential.
2. The Nkana area is thermally immature with TOC values up to 0.62wt%. This suggests that this area has poor hydrocarbon source potential.
3. The Amuvi area is thermally immature with TOC values up to 0.78wt% suggesting a poor hydrocarbon source potential.
4. The Okobo area member samples vary from immature to mature. It contains TOC up to 0.71wt%. Therefore, it has poor-fair hydrocarbon source potential.
5. The Obotme area member samples vary from immature to peak mature. It has TOC values up to 0.88wt% which suggests a fair hydrocarbon generation potential in the area that is mature.
6. There is maturity variation between the two formations (Nsukka and Mamu) which may have resulted from the inferred fault as seen on the geologic map of the study area.

ACKNOWLEDGEMENT: The authors wish to acknowledge the effort of staff of Civil Engineering and Geosciences Laboratory, Newcastle University and Plant Anatomy and Physiology Research Laboratory, University of Port Harcourt during the course of analysis of samples for Rock-Eval pyrolysis and Soluble Organic matter respectively.

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