



## Integrated analysis Rock-Eval pyrolysis and Vitrinite Reflectance data for organic matter of the Fula Sub-Basin, Muglad Basin, Sudan

Sadam H M A Eltayib

Department of Geology, Faculty of Science and Technology-Omdurman, Omdurman Islamic University, Khartoum, Sudan

### Abstract

In this study, Integrated Analysis Rock-Eval pyrolysis and Vitrinite Reflectance data for organic matter of the Fula Sub-Basin, Muglad Basin, Sudan. The Total Organic Carbon (TOC %) and pyrolysis data are used to determine the source rock quality and quantity as organic richness, source rock generating capability and kerogen types of the source rock. The vitrinite reflectance measurements ( $R_o$  %) of some selected samples, which are the most powerful paleogeothermometer maturity indicator are used to detect the thermal maturity level of source rocks. The TOC content of the analyzed samples varies from 0.29% to 4.92% with an average of 3.01% in studied horizon. In general, based on TOC values, the studied samples denoting fair to very good source rocks. The thermal maturity of the organic matter in shales of FULA-N-21 Well source rock is determined in this study using the vitrinite reflectance  $R_o$  (%) and the pyrolysis results HI and Tmax. Vitrinite reflectance measurements ( $R_o$ , wt. %) of shales of FULA-N-21 Well range from 0.69 to 0.93% indicating mature organic matter at the depth value 2045 m down to 3270 m. The maturation of organic matter stage was marked by a significant decrease of oxygen content of kerogen and a correlative increase in carbon content with increasing depth. It results in a slight decrease of H/C and a marked decrease of O/C ratios. The vitrinite reflectances ( $R_o$ ) are usually less than 0.5% in the catagenesis stage.

**Keywords:** Pyrolysis, Rock-Eval, organic matter, source rock

### Introduction

The Fula sub-basin is a fault-bounded, asymmetric half-graben located in the NE of the Muglad Basin, and is up to 120 km long and 41 km wide covering an area of 3560 km<sup>2</sup> (Fig. 2). The sub-basin is separated from the northern part of the Kaikang Trough by the Babanusa Uplift (Fig. 1). Petroleum exploration in the sub-basin began in 1974 but only one wildcat well (Baleela-1, 1985) was drilled, recording very weak oil or gas shows; little further exploration work was undertaken during subsequent decades.

However a renewed exploration programme including 2000 km of 2D seismic, 1334 km<sup>2</sup> of 3D seismic and the drilling of 68 exploration wells has been carried out since 1996. Eleven commercial and non-commercial oilfields (Fula, Fula North, Fula Northeast, Fula South, Fula West, Moga, Keyi, Jake, Jake South, Bara and Arad) have been discovered in the sub-basin, and oil production is currently about 60,000 barrels per day. In this study, we integrate new source and reservoir rock analytical data for the Fula sub-basin into the regional framework of the Muglad Basin, and investigate the differences between petroleum accumulations in the Fula sub-basin and those elsewhere in the Muglad Basin. The sedimentary column in the Fula sub-basin is up to 8400 m thick. Based on an interpretation of seismic data and drilling results, the succession can be divided into four sequences (Fig. 2) separated by unconformities and related conformities: Lower Cretaceous, Upper Cretaceous, Paleogene and Neogene – Quaternary.

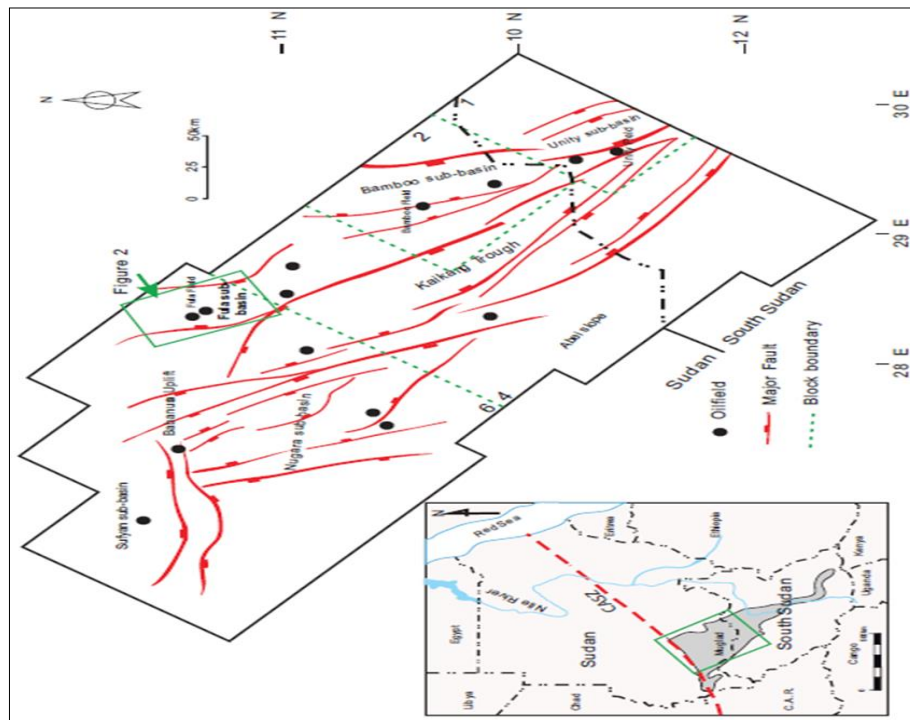
### Objectives

The main objectives of this study are:

- The evaluation of the source-rock parameters of studied intervals.
- Developing and measuring the maturity history of source rocks in the area.
- Determination of the hydrocarbon generative potential of source rocks.

### Materials and Methods

Source rock and reservoir rock samples were analyzed at the Central Petroleum Laboratories (CPL) of the Ministry of Petroleum and Gas, Khartoum, Sudan. Rock-Eval pyrolysis is used extensively for characterizing the quality, quantity and thermal maturity of organic matter in sedimentary rocks, parameters that are essential for assessing the petroleum potential of sedimentary basins. Source rock TOC was measured using a Leco Model CS-400 instrument. Seventeen samples were screened by Rock-Eval pyrolysis using a Vinci Model 2-plus with standard operational conditions. Cuttings samples were extracted with chloroform using a Soxhlet apparatus for 20 hours. Separation and quantification of bitumen was performed using an Iatroscan Model 5-new to obtain the saturate, aromatic, resin and asphaltene fractions. Vitrinite reflectance is a well established and widely used thermal maturity parameter for evaluating the petroleum potential of sedimentary basins. Eight whole rock cuttings and conventional core samples were prepared for organic petrology and vitrinite reflectance analysis.



**Fig 1:** Main map shows structural units in the Muglad Basin, which extends from the Republic of Sudan to the Republic of South Sudan, with the location of the Fula sub-basin and the main oilfields so far discovered. The smaller map shows the regional location of the Muglad Basin. Key: CASZ = Central African shear zone; C.A.R. = Central African Republic.

| Age                          | Units & Formations                                                                  |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   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**Fig 2:** Generalised stratigraphic column for the Fula sub-basin showing lithologies, source, reservoir and seal units and summary of the three syn-rift stages mentioned in the text (modified from Schull, 1988<sup>[41]</sup>; McHargue *et al.*, 1992; Tong *et al.*, 2004)<sup>[49]</sup>. Rift cycles are Early Cretaceous, Late Cretaceous and Paleogene. The main source rocks were deposited during the first syn-rift stage. Commercial oil reserves have been found in the Abu Gabra, Bentiu and Aradeiba Formations.

## Results and Dissection

### 1. Rock Eval Pyrolysis and Total Organic Carbon Content (TOC) Determination

The natural thermal cracking of kerogen in source rocks can be carried out in laboratories by using a certain analysis is termed pyrolysis. Pyrolysis is probably the best routine tool for determining the parameters required to define the kerogen type, Generation Potential (GP), Transformation Ratio (TR) and also to predict the nature and quantity of hydrocarbon that could be generated from kerogen. The most common technique of this process is named Rock-Eval pyrolysis (Espitalié *et al.*, 1985) [16]. During the slow heating of a rock-sample, two slugs of hydrocarbon are released from the rock. The first slug is named S1 which come slightly above 250 °C. The S1 represents the hydrocarbon already present in the rock and appears as a peak on the monitor of the Rock Eval pyrolysis. This peak decreases with increasing temperature. As heating continues, a second slug of hydrocarbon come out within a range of temperature between 420 °C–460 °C, called S2 and decreases as heating continues. S2 represents the hydrocarbon generated from the kerogen by thermal decomposition and represents the amount in milligram of hydrocarbon per gram of rock. During the pyrolysis process, CO<sub>2</sub> is released from the kerogen and trapped into second detector giving a third peak named S3. S3 is used as an indicator for the amount of oxygen present in the kerogen. The Rock-Eval pyrolysis and the TOC content were determined by using the Rock-Eval 6 Standard equipment. About 70-100mg of pulverized sample is place in the crucible and is progressively heated to 650°C under an inert atmosphere (pyrolysis oven). During the analysis, the hydrocarbons that already present in the sample are volatilized at a moderate temperature. The amounts of hydrocarbons are measured and recorded as peak known as S1. Next pyrolyzed is the kerogen present in the sample, which generates hydrocarbons and hydrocarbon like compounds (recorded as S2 peak), CO<sub>2</sub> and water. The CO<sub>2</sub> generated is recorded as the S3 peak. At oxidation oven sample is heated up to 850°C where residual carbon is measured and recorded as S4.

These results of Rock-Eval pyrolysis technique is reported as raw data S1, S2 and S3 in milligram of hydrocarbon or CO<sub>2</sub> per gram of rock and then normalized for the organic carbon content of the sample, yielding values in milligrams of hydrocarbon per gram of TOC. The normalized values of S2 are named the hydrogen index (HI) which is used to define the kerogen type, and S3 is named oxygen index (OI). Plotting HI versus OI leads almost to the same results given by the elemental analysis data (H/C and O/C atomic ratios) on a Van Krevelen diagram. Pyrolysis gives also the S2 peak temperature (Tmax) indicating the temperature at which the maximum pyrolytic hydrocarbons liberated. Based on the data of S1, S2 taken from Rock Eval Pyrolysis. The percent TOC is not measured directly, but calculated from the formula:

$$\% \text{TOC} = 0.082[(S1 + S2) + S4] / 10$$

The Rock-Eval pyrolysis instrument was developed by the Institute Francais du Petrole. It provides a rapid semi quantitative source rock analysis on small sample of rock by heating it, using a special temperature program. It is an excellent screening technique for identifying possible source and reservoir intervals on which more detailed analysis should be performed. Information generated by the device include:

### 2. Free Hydrocarbon (S1)

The S1 represents free hydrocarbons in rock samples before the analysis. When S1 is large relative to S2, an alternative source such as migrated hydrocarbons or contamination should be suspected. The readings of S1 in the studied samples range from 0.05 – 5.21 ppm, with the average concentration 1.46 (mgCO<sub>2</sub>/g rock) Fig. 3.

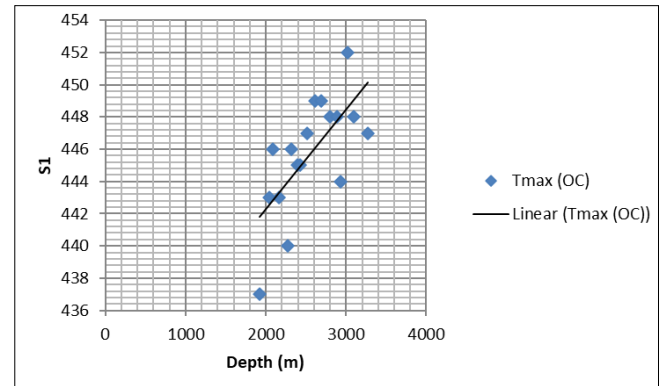


Fig 3: The relationship between S1 values and depth

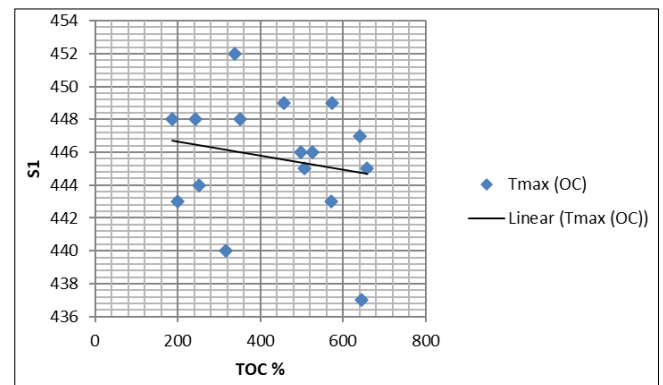


Fig 4: The relationship between TOC values and S1 (mg HC/g rock)

### 3. Hydrocarbon Generating Potential (S2)

The S2 values represent the amount of hydrocarbon that formed during thermal pyrolysis of the sample. It can either represent the total hydrocarbon generating potential for immature samples or residual hydrocarbon generating potential for mature and post-mature sample. The readings of S2 in the studied samples range from 0.59 – 32.35 (mgCO<sub>2</sub>/g rock), with the average concentration 14.12 (mgCO<sub>2</sub>/g rock) Fig. 5. All analyzed samples are within the good source rock potential limits according to the classification of Peters, 1986 [37].

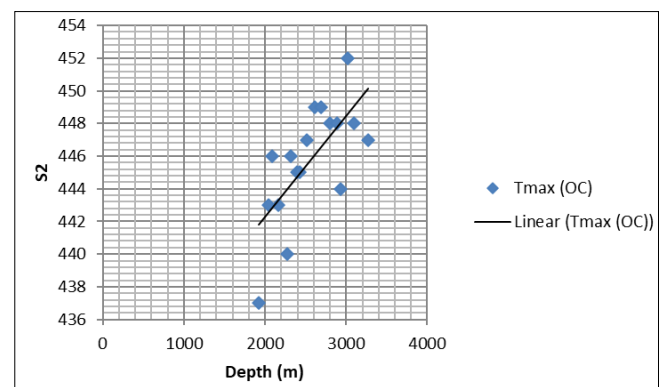


Fig 5: The relationship between S2 values and depth

#### 4. Hydrogen Index (HI)

The hydrogen index (HI) represents the amount of hydrogen relative to the amount of Organic carbon present in a sample. It is defined as:

$$(S_2/TOC) \times 100$$

This index is used to determine the type of organic matter and whether the organic matter is currently oil- prone or gas- prone. The type of organic matter can be determined by plotting HI versus Tmax. Low HI values (100-200 mg S<sub>2</sub>/ g TOC) are characteristic of Type III (gas- prone) organic matter and high HI values (> 300mg S<sub>2</sub>/ g TOC) are characteristic of Type II and Type I (oil- prone) organic matter. The readings of HI in the studied samples range from 64 – 657 (mg S<sub>2</sub>/g TOC), with the average concentration 407.4 (mg S<sub>2</sub>/g TOC) Fig. 6. All analyzed samples are within the Oil prone limits according to the classification of Peters, 1986<sup>[37]</sup>.

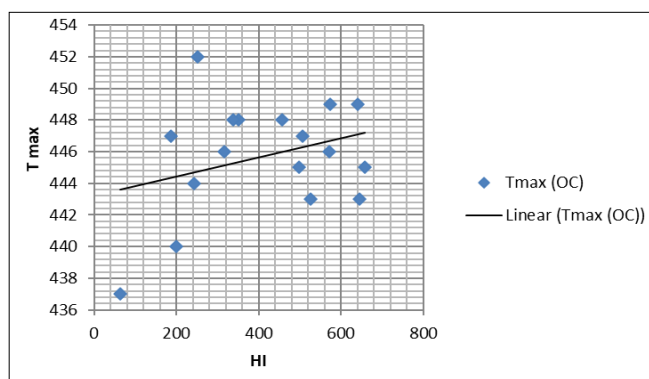


Fig 6: The relationship between HI values and Tmax (C°)

#### 5. Maximum Pyrolysis Temperature (Tmax)

Tmax is the temperature at which the maximum rate of hydrocarbon generation occurs in a kerogen sample during pyrolysis (the maximum temperature of the S<sub>2</sub> peak). It is related to the degree of thermal maturation of the sample analysed. The Tmax value cannot be used in interpretation when the S<sub>2</sub> peak is small, broadened or multi-modal. The readings of T<sub>max</sub> in the studied samples range from 437 – 452 (°C) Fig. 7, with the average concentration 445.6 (°C). All analyzed samples are within the Oil (from type III kerogen) limits according to the classification of Peters, 1986<sup>[37]</sup>. The study provided also the relationship between pyrolysis Hydrogen Index (HI) and peak temperature of pyrolysis (T<sub>max</sub>) of source rock in the study area which shows that the organic type in the four horizons are mainly of Type III. Based on the analysis of the relationship between HI and TOC, it is noted that the HI of most of the

samples which show Type II2 has a relation with the higher content of TOC, so in general the organic type is mainly Type III, Fig. 8.

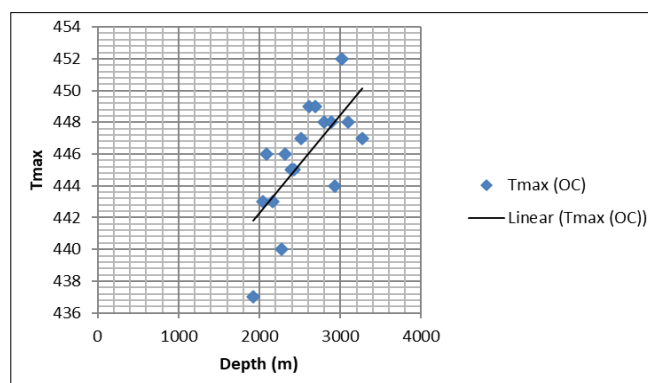


Fig. 7: The relationship between T<sub>max</sub> values and depth

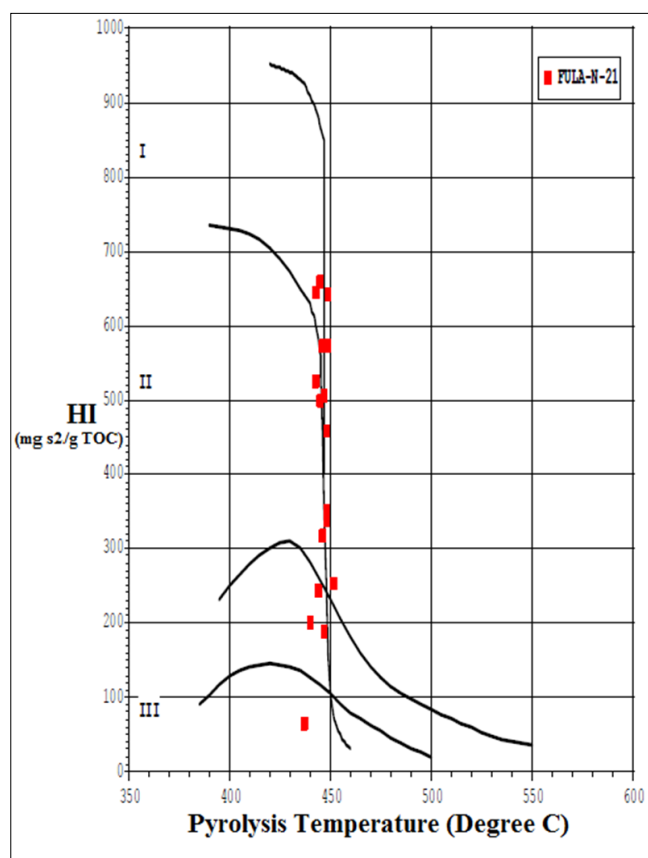


Fig 8: The relationship between HI values and pyrolysis

Table 1: Geochemical Data (Rock-Eval Pyrolysis) of of FULA-N-21 Well

| NO | Depth (m) | TOC (%) | S1 (mgCO <sub>2</sub> /g rock) | S2 (mgCO <sub>2</sub> /g rock) | S3 (mgCO <sub>2</sub> /g rock) | S1+S2 (mgHC/g) | Tmax (°C) | HI  | OI | TPI S1/S1+S2 |
|----|-----------|---------|--------------------------------|--------------------------------|--------------------------------|----------------|-----------|-----|----|--------------|
| 1  | 1925      | 0.92    | 0.05                           | 0.59                           | 0.78                           | 0.64           | 437       | 64  | 84 | 0.07         |
| 2  | 2045      | 3.96    | 0.69                           | 25.51                          | 0.79                           | 26.20          | 443       | 644 | 20 | 0.03         |
| 3  | 2090      | 3.03    | 0.89                           | 17.34                          | 0.67                           | 18.23          | 446       | 571 | 22 | 0.05         |
| 4  | 2165      | 2.49    | 0.22                           | 13.06                          | 0.66                           | 13.28          | 443       | 525 | 26 | 0.02         |
| 5  | 2270      | 1.72    | 0.63                           | 3.44                           | 0.74                           | 4.07           | 440       | 200 | 43 | 0.16         |
| 6  | 2320      | 1.78    | 0.34                           | 5.66                           | 0.63                           | 6.00           | 446       | 317 | 35 | 0.06         |
| 7  | 2390      | 3.47    | 0.56                           | 17.32                          | 0.75                           | 17.88          | 445       | 498 | 22 | 0.03         |
| 8  | 2425      | 4.92    | 1.08                           | 32.35                          | 0.74                           | 33.43          | 445       | 657 | 15 | 0.03         |
| 9  | 2510      | 2.52    | 0.24                           | 12.73                          | 0.71                           | 12.97          | 447       | 506 | 28 | 0.02         |
| 10 | 2610      | 4.20    | 0.66                           | 26.94                          | 0.72                           | 27.60          | 449       | 641 | 17 | 0.02         |
| 11 | 2685      | 4.04    | 0.58                           | 23.15                          | 0.81                           | 23.73          | 449       | 573 | 20 | 0.02         |
| 12 | 2795      | 4.32    | 2.69                           | 19.74                          | 0.77                           | 22.43          | 448       | 457 | 18 | 0.12         |

|    |      |      |      |       |      |       |     |     |    |      |
|----|------|------|------|-------|------|-------|-----|-----|----|------|
| 13 | 2890 | 3.00 | 2.00 | 10.51 | 0.53 | 12.51 | 448 | 350 | 18 | 0.16 |
| 14 | 2930 | 2.95 | 2.87 | 7.14  | 1.48 | 10.01 | 444 | 242 | 50 | 0.29 |
| 15 | 3015 | 2.55 | 2.54 | 6.40  | 0.47 | 8.94  | 452 | 251 | 18 | 0.28 |
| 16 | 3100 | 2.29 | 1.12 | 7.74  | 0.69 | 8.86  | 448 | 337 | 30 | 0.13 |
| 17 | 3270 | 3.03 | 5.21 | 5.68  | 0.97 | 10.89 | 447 | 187 | 32 | 0.48 |

## 6. Vitrinite Reflectance Measurements

This technique was developed since long time ago to be used for measuring the coalification rank, in which the vitrinite macerals is usually very common. This method is based on the fact that with increasing the thermal stress, the reflection value of vitrinite increases. Tissot and Welte (1978)<sup>[44]</sup> considered Ro% as the most powerful maturation-measuring tool. Furthermore, Ro% is not dependent on kerogen type, rock lithology, or contamination, as many other maturation parameters. However, in case of type I kerogen, because of its deficiency in vitrinite macerals, a large amount of samples would be required for microscopic examination in order to measure statistically significant number of vitrinite grains. Whole rock samples were crushed to about 1mm diameter and mounted on resin blocks. The harden block were ground using silicon carbide paper and polished with diamond paste of various grades. Vitrinite reflectance measurement were made using J&M MPM 200 photometer microscope under oil immersion at a wavelength of 546 nm. Prior to the analysis, the photometer was calibrated using (saphir) standard of 0.591% reflectance and checked by (gadolinium-gallium-granat) standard of 1.718% reflectance. The evolution pathways and maturation stages indicate that temperature and time play the main role in forming hydrocarbons in source rocks. The depth interval in which a petroleum source rock generates and expels most of its oil is called the oil window.

## 7. Total Organic Carbon Content (TOC)

The amount of organic matter buried and preserved in the rock is expressed by the total organic carbon content (TOC) values in weight percent (wt.%) of the dry rock. TOC values express the organic richness of sediment but not the hydrocarbon potential, as a significant proportion of the TOC may represent inertinite, which has no hydrocarbon

potential at any level of maturity. The measured TOC value is not indicative of the sample's source potential. Vitrinite reflectance measurements record the percentage of incident light, which is reflected by individual vitrinite particles. The measurements are made on a rock sample embedded in a polished plastic plug from which individual vitrinite particles are identified. A large number of particles are analyzed in each sample and the results are reported in histogram form with an accompanying statistical analysis. Vitrinite reflectance increases regularly with increasing thermal alteration. Therefore, the degree of thermal maturity of the sediment can be determined. One of the requirements needed for source rocks to generate commercial amounts of oil is that, they must contain sufficient quantity of organic matter quite enough to generate and expel hydrocarbon. The Total Organic Carbon (TOC %) and pyrolysis data are used to determine the source rock quality and quantity as organic richness, source rock generating capability and kerogen types of the source rock. The vitrinite reflectance measurements (Ro %) of some selected samples, which are the most powerful paleogeothermometer maturity indicator are used to detect the thermal maturity level of source rocks. The total organic carbon analysis measures the organic richness of a rock in weight percent organic carbon. It is an excellent screening technique to determine which samples are worthy of further examination. A total organic carbon content of 0.5 % TOC for shale and 0.3% TOC for carbonates are generally considered the lower limit for effective source rocks. The TOC content of the analyzed samples varies from 0.29% to 4.92% with an average of 3.01% in studied horizon Fig. 9. In general, based on TOC values, the studied samples denoting fair to very good source rocks according to the classification of Peters, 1986<sup>[37]</sup>.

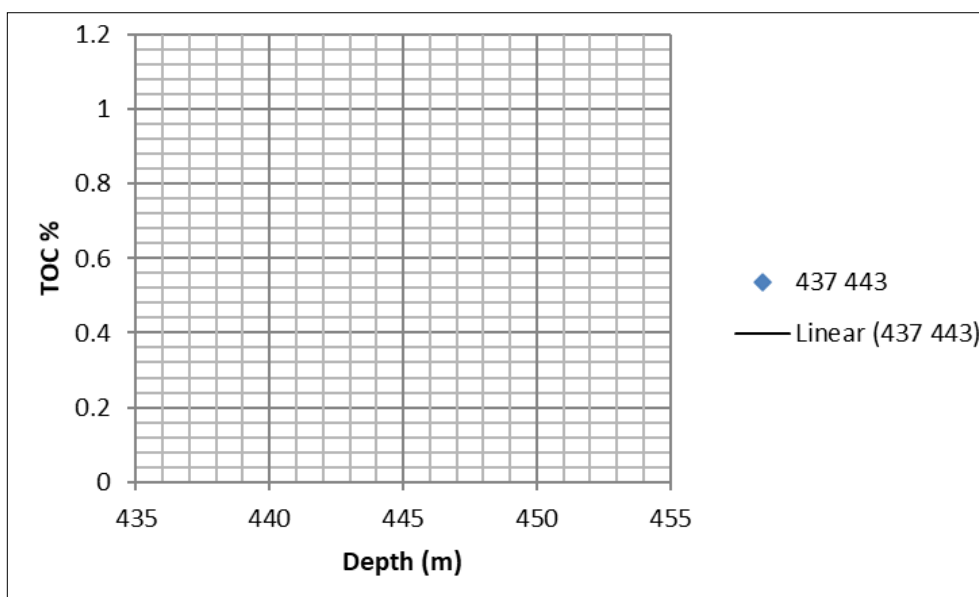
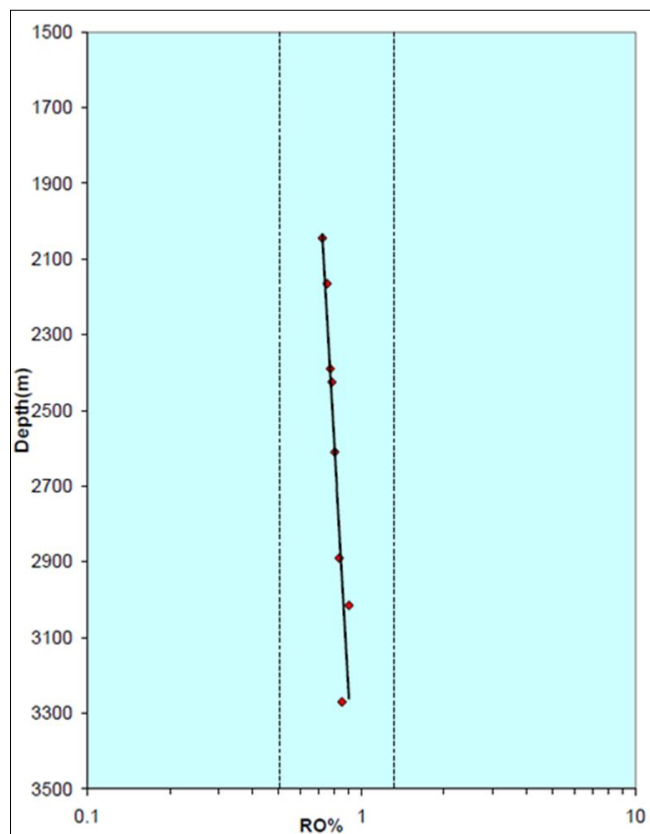


Fig 9: Relationships between TOC and Depth values.

## 8. Thermal maturity of the organic matter

The thermal maturity of the organic matter in shales of FULA-N-21 Well source rock is determined in this study using the vitrinite reflectance  $R_o$  (%) and the pyrolysis results HI and  $T_{max}$  cited in the previous studies on the area. Vitrinite reflectance measurements ( $R_o$ , wt. %) of shales of FULA-N-21 Well range from 0.69 to 0.93% indicating mature organic matter at the depth value 2045 m down to 3270 m (Table 2, Fig. 10). The relation between  $R_o$  values and depth of samples shows that the roof of the oil generating window lies at depth 1750m while that of the gas window is at depth 3550m. The floor of the gas window lies at depth 3070m. Deeper than 4500m, the organic matter will arrive to over maturity stage. Many samples in studied horizon at shallow depths show higher  $R_o$  than deeper ones, which may be due to the tectonic uplift. In addition, pyrolysis  $T_{max}$  values of analyzed samples reflect mature to immature stage for the studied rocks. In general,  $T_{max}$  maturity pattern differs slightly than that given by the  $R_o$  values.



**Fig 10:** Relationship between Vitrinite reflectances ( $R_o$ ,%) and depth

**Table 2:** Geochemical Data (Vitrinite Reflectance VRO. Data) of FULA-N-21 Well

| No. | Sample Depth (m) | V Ro. Readings |         |          | No. of readings |
|-----|------------------|----------------|---------|----------|-----------------|
|     |                  | Min (%)        | Max (%) | Mean (%) |                 |
| 1   | 2045             | 0.64           | 0.80    | 0.72     | 12              |
| 2   | 2165             | 0.62           | 0.83    | 0.75     | 18              |
| 3   | 2390             | 0.63           | 0.87    | 0.77     | 21              |
| 4   | 2425             | 0.67           | 0.90    | 0.78     | 16              |
| 5   | 2610             | 0.70           | 0.98    | 0.80     | 14              |
| 6   | 2890             | 0.75           | 0.98    | 0.83     | 16              |
| 7   | 3015             | 0.78           | 1.07    | 0.90     | 11              |
| 8   | 3270             | 0.70           | 1.02    | 0.85     | 16              |

## Conclusions

The type of organic matter complements the organic richness in evaluating the generating potential of the source rock. The different types of organic matter can be identified in a kerogen and thus be related to its origin. The kerogen is defined as all the disseminated organic matter in sedimentary rocks of very complicated chemical structures, that insoluble in organic or non organic solvents. The different types of organic matter can be identified in a kerogen and thus be related to its origin. Kerogen is subjected to physical and chemical changes due to increasing temperatures and pressures as the depth of burial increase. The result of these changes is the generation of hydrocarbons. Maturation can be defined as the physicochemical transformation of Organic Matter during the burial history of source rocks in sedimentary basins. Maturation of Organic Matter in a source rock is controlled by biological activity in an early stage, then physical and chemical effects due to elevated temperatures and pressures. Thus, maturity is a product of the combined effect of elevated temperature due to progressive depth of burial and geologic age of rocks.

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