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VARIOUS TECHNIQUES TO EVALUATE CARBONACEOUS SOURCE ROCKS AS AN UNCONVENTIONAL GAS RESERVOIR

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ABSTRACT

Organic geochemical data of 17 outcrops which were collected in underground mines of Jammu regions of Northwest Himalaya, India was obtained from a published research paper (Mani *et al*., 2014) in order to study the potential of source rock as an unconventional gas reservoir. The data was obtained by using TOC/Rock-Eval pyrolysis and vitrinite reflectance. TOC/Rock-Eval pyrolysis is the most basic technique used in source rock evaluation. This project involved a case study to learn the techniques to evaluate source rock by using data obtained from various techniques. TOC/Rock-Eval pyrolysis data shows that the total organic carbon (TOC W%) of Eocene Subathu shales ranged from 2.42 to 30.4%. High TOC value showing that Eocene Subathu shales are good quality source rock with high organic content. Besides that, the values of free hydrocarbon in the source rock (S1) and those hydrocarbon generating through thermal cracking (S2) also observed to be high (S1 ranged from 0.1 to 2.66 and S2 ranged from 0.51 to 15.54 mg HC/g rock). In terms of thermal maturity, the value of T_{max} obtained from Rock-Eval pyrolysis observed to be high (T_{max}> 4500C for all samples) and vitrinite reflectance also showing a high Ro value (R_o>1.5%) indicates that Eocene Subathu shales are post-mature source rock and falls at a dry gas stage. According to HI and OI correlation, organic matter in Eocene Subathu shales are showing characteristics of an over-mature gas prone Type III kerogen with fair to excellence gas generating potential.

Keywords: unconventional gas reservoir, source ocks, virtrinite reflectance, thermal maturity.

INTRODUCTION

A great amount of natural gas mostly methane gas which is trapped in an organic-rich, fine-grain sedimentary rocks has rapidly emerged as an unconventional energy source globally in recent years. (Boyer *et al*., 2006). Unconventional shale gas is generated from the organic matter trapped in the fine-grain sediments under the effect of heat and pressure in the subsurface. Greg *et al*. (2012) mentioned that unconventional resources found on our planet have a higher potential to produce more oil and gas as compared to conventional resources and they were the main target of exploration and production orders for the past 150 years. With the advance in exploration technologies such as horizontal drilling and hydraulic fracturing have made the effort to extract hydrocarbons from unconventional reservoir possible and more economical as compared to conventional drilling. IEA-USA predicts that the global natural gas reserves can last for 250 years with current consumption rate with the reserve addition from shale. Hence, the study on unconventional gas reservoir has become significant globally.

Type of hydrocarbon produced from a source rock depends on three important parameters which are the type of organic matter (kerogen) in the source rock, organic richness, and its subsequent thermal evolution or thermal maturity (Espitalie, Madec, Tissot, Mennig, & Leplat, 1977). The organic matter in shales will transform into an insoluble organic matter known as kerogen under heat and pressure during sedimentation. The kerogen will continually transform into a different type of hydrocarbon provided sufficient heat and organic richness is available (McCarthy *et al*., 2011).

Various source rock evaluation techniques have been developed in order to identify the type of kerogen, organic richness and thermal maturation of a source rock. However, TOC/Rock-Eval pyrolysis is the most basic and reliable rock evaluation techniques used to characterize hydrocarbon generating potential of a source rock because it allows the rapid evaluation to be carried out on small quantities of rock (McCarthy *et al*., 2011). Pyrolysis can be defined as a method of downgrading the organic matter in rock by heating them up to a different programmed temperature in order to identify the composition of hydrocarbons in the source rock (K. Peters, 1986).

In TOC/Rock-Eval pyrolysis technique, the hydrocarbon and carbon dioxide gas that released during steady heating will be measured by flame ionization detector (FID) and infrared detector whereby a thermocouple will be used to monitor the temperature setting of the equipment (Nunez-Betelu & Baceta, 1994). Vitrinite reflectance was used as an additional diagnostic tool for assessing maturation. It is used to determine thermal maturation of a source rock by measuring the reflectivity of oil (R_o) (McCarthy *et al*., 2011). Important parameters such as S1, S2, S3, T_{max}, Hydrogen index (HI), Oxygen index (OI) and vitrinite reflectivity (R_o) obtained from both techniques will be used for characterizing the source rock hydrocarbon generation potential.

In this project, data was obtained from a published journal conducted by Mani *et al.* (2014), on Eocene Subathu shales collected from Jammu regions of Northwest Himalaya. 17 shale samples have been selected out of 64 samples for study purpose. Organic richness, type of kerogen and thermal maturity of Eocene Subathu shales can provide useful information for the evaluation of gas generating potential of a source rock.

A. GEOCHEMICAL ATTRIBUTES FOR SOURCE ROCK EVALUATION

Organic richness

Hydrocarbons are generated only if the source rock contains sufficient amount of organic matter in it (Nunez-Betelu & Baceta, 1994). Without sufficient amount of organic matter, the possibility for a source rock to generate hydrocarbon is nearly zero. Shales which is made up of fine-grained sediments mostly are organic-rich as fine grain pores can prevent the organic matter from losing through oxidation and micro-biogenic activity. Organic richness can be described as the amount of organic matter preserved in the source rock and it can be measured by using TOC (Total Organic Carbon) content. TOC content was obtained from the sum of pyrolyzed carbon and residual carbon through Rock-Eval pyrolysis. Pyrolyzed carbon is carbon which has been losing as hydrocarbon during the heating process and residual carbon is the spent hydrocarbon which has nil hydrocarbon generation potential even undergo further heating. Organic richness varies according to lithology. For shales, TOC value which is higher than 2% and above usually are good quality source rock but for limestone even a lower value of TOC are good source rock (Nunez-Betelu & Baceta, 1994).

Types of organic matter

Kerogens are an insoluble organic matter present in sedimentary rocks. The lack of solubility in the organic solvent is because of its large molecular weight (Dow, 1977). Kerogens are an important element which can affect hydrocarbon generation potential of a source rock. There are four different type of kerogen namely Type I, Type II, Type III, and Type IV kerogen. Each type of kerogen is varied in terms of their depositional environment and precursor. These causes each type of kerogen to generate a different type of hydrocarbon in a source rock. Hydrogen index (H:C ratio) obtained from Rock-Eval pyrolysis can be used to differentiate the different type of kerogen. Type I kerogen usually gives the highest HI values (Nunez-Betelu & Baceta, 1994). It derived mainly from algae which are organic rich and it usually contain high HI value (HI>300) (McCarthy *et al*., 2011). A mature source rock with Type I kerogen usually will generate oil. Type II kerogen has high HI value but not as high as Type I kerogen. (Nunez-Betelu &Baceta, 1994). It derived mainly from the mixture of plankton and some algae that usually generate both oil and gas provided that the source rock achieves sufficient maturation and contain sufficient organic richness. Type II kerogen can be identified by its HI value (200<HI<300) (McCarthy *et al*., 2011). Type III kerogen which mainly derived from terrestrial plants will contain lower HI value as compared to Type I and Type II (50<HI<200) and a Type III source rock mostly are gas-prone. Whereby a Type IV kerogen has no hydrocarbon generation potential because the hydrogen in the organic matter has been lost due to oxidation. Type IV kerogen contains only graphite which is made up of carbon residue. Table-1 and Table-2 below shows the classifications of different type of kerogen and classifications of different type of kerogen based on hydrogen index (HI).

Table-1. Classification of kerogen types (McCarthy *et al.,* 2011).

Kerogen Type	Source Material	Depositional Environment
I	Mainly algae	Lacustrine setting
II	Mainly plankton and some contribution from algae	Marine setting
III	Mainly terrestrial plants	Terrestrial setting
IV	Reworked, oxidized material	Varied setting

Table-2. Classification of kerogen types based on hydrogen index (HI) (McCarthy *et al.,* 2011).

Kerogen Type	Product Type	Hydrogen Index
I	Gas	>300
II	Gas and Oil	200 to 300
III	Oil	50 to 200

Thermal maturity

Thermal maturation is one of the significant parameters which can affect the hydrocarbon generation potential of a source rock. The maturation pathway of source rock can be divided into three main stages which are diagenesis, catagenesis, and metagenesis.

Diagenesis can be described as the stage where the organic matters fall in the immature zone. If source rock was deposited under an anoxic environment during diagenesis, dry gas may be formed from the existing organic matter due to the activity of methanogenic bacteria. With the increase in burial depth, temperatures and changes in pH, the organic matter will slowly transform into insoluble kerogen and a little amount of soluble bituminous products (North, F.K., 1985).

Catagenesis is the stage where kerogens fall in the maturation zone. During this stage, hydrocarbons will be generated as temperature increases range from 50-150 degree Celsius (Kevin McCarthy, 2011). This ranges from temperature also known as “oil window”. The type of hydrocarbons generated at this stage will be much dependent on the type of kerogen in the source rock. Type I and Type II kerogens produce both oil and gas during catagenesis whereby a Type III kerogen will produce mostly hydrocarbon gas if it falls at the mature zone. Wet gas may be produced through thermal cracking of oil molecules formed if Type III kerogens undergo further heating.

Metagenesis usually happens at high temperature which ranges from 150-200 degree Celsius in subsurface temperature. At this stage, the additional heat will convert most of the kerogen into dry gas and carbon residual which is graphite. Source rock at this stage is known as “over

mature source rock". As the heat continously increasing in the gas window, dry gas and other forms of gas such as carbon dioxide, nitrogen or hydrogen sulfide may be evolved from the source rock. Usually, if the source rock is found to be over mature, it indicates that the source rock had undergone much alteration process and it has exhausted all hydrocarbon generation potential to produce any further oil or gas (North, F.K., 1985).

Thermal maturation can be determined by using T_{max} value from TOC/Rock-Eval pyrolysis and R_o from vitrinite reflectance. T_{max} can be defined as pyrolysis temperature at which maximum amount of hydrocarbon from the thermal degradation of kerogen is generated (Espitalié, 1986). It is the temperature of S2 peak in pyrogram. Maturation of source rock will increase relative to the increase in T_{max}. A source rock with T_{max} less than 435 ˚C are at the immature zone, T_{max} ranged from 435-455 ˚C are at the mature zone, T_{max} of 455-470 ˚C is transitional zone and T_{max} higher than 470 ˚C are considered as an over-mature zone which is metagenesis (Nunez-Betelu & Baceta, 1994). Type of hydrocarbons produced will be depending on the T_{max} value and type of kerogen present in the rock. Whereas in vitrinite reflectance method, the value of R_o is used to evaluate the thermal maturation of a source rock. The higher the reflectivity of maceral, the higher the maturity of a source rock. Figure-1 below illustrates the process of thermal maturation of source rocks.

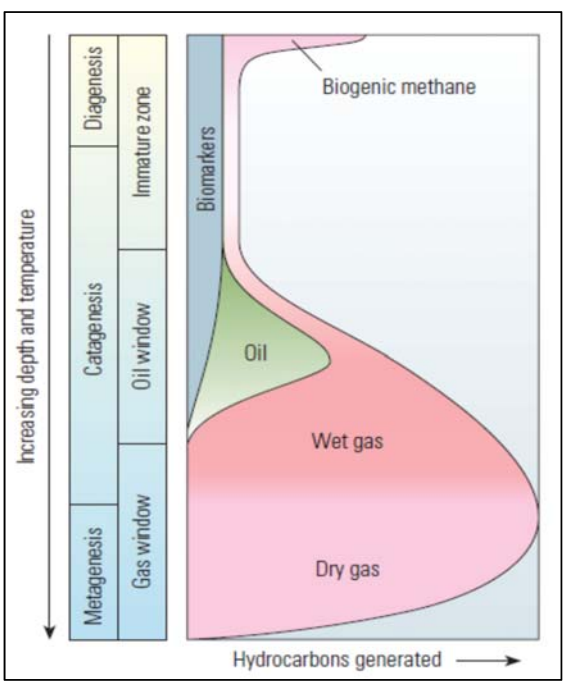


Figure-1. Thermal maturation of source rock (McCarthy *et al.,* 2011).

B. SOURCE ROCK EVALUATION TECHNIQUE

Total organic carbon method (TOC)

Total Organic Carbon method is a priority in source rock assessment. Rock-Eval pyrolysis and vitrinite reflectance usually will be used as screening procedure after TOC. TOC is important in source rock analysis because hydrocarbons are derived mainly from carbon and hydrogen. Without sufficient amount of carbon content or organic richness, there will be zero possibility for a source rock to produce hydrocarbons. In order to determine the organic richness, TOC will be applied to measure the carbon content in the rock sample. According to McCarthy *et al*., (2011), TOC values are obtained through direct combustion of 1g samples of rock. The rock sample will be heated at 1200 degree Celsius with the use of high-frequency induction furnace. Carbon which has been evolved during combustion will be measured by an infrared cell and will be converted to TOC as weight percent of the rock. TOC also can be obtained directly from Rock-Eval pyrolysis by using the data of pyrolyzed carbon and residual carbon (Mani et.al. 2014).

Rock-Eval pyrolysis techniques

Rock-Eval pyrolysis technique is implied to identify the hydrocarbon generating the potential of source rock and the thermal maturity of source rock (Peters, 1986). Rock-Eval pyrolysis method is the most basic organic geochemical analysis of sedimentary organic matter and it is commonly used in analyzing rock samples (Bajor *et al*., 1969). Pyrolysis is a method of downgrading the organic matter in rock by heating them up to a different programmed temperature in order to identify the composition of hydrocarbons in the source rock (K. E. Peters & Cassa, 1994). Rock-Eval pyrolysis technique uses a programmed temperature that is parallel to the subsurface temperature in order to obtain results which took millions of year in a sedimentary basin (McCarthy *et al*., 2011). There are four important parameters that are useful for source rock evaluation. There are S1 (free hydrocarbon that has been generated in the source rock), S2 (amount of potential hydrocarbon that can be generated through thermal cracking of kerogens), S3 (amount of carbon dioxide present in the rock) and T_{max} (temperature which correspond to maximum generation of hydrocarbon or temperature of S2 peak) (McCarthy *et al*., 2011). Then S2 and S3 can be used to generate hydrogen index (HI) and oxygen index (OI) to determine the type of kerogen whereby T_{max} can be used to determine the thermal maturity of source rocks.

Vitrinite reflectance

Vitrinite reflectance can be used as an additional diagnostic tool for assessing maturation of source rock. As mentioned by McCarthy *et al*., (2011). Vitrinite reflectance was first used in assessing the thermal maturation for coal but now it is used to evaluate the thermal maturity of kerogen over temperatures. This technique involved the measure of the percentage of incident light reflected from the surface of vitrinite particles in a sedimentary rock. The percentages of reflected light will be recorded in term of percentage R_o. Results are often presented as a mean R_o value based on all vitrinite particles measured in an individual sample. The thermal maturity of source rock can be determined based on the range of R_o that obtained from

this technique. Figure-2 shows the vitrinite reflectance method.

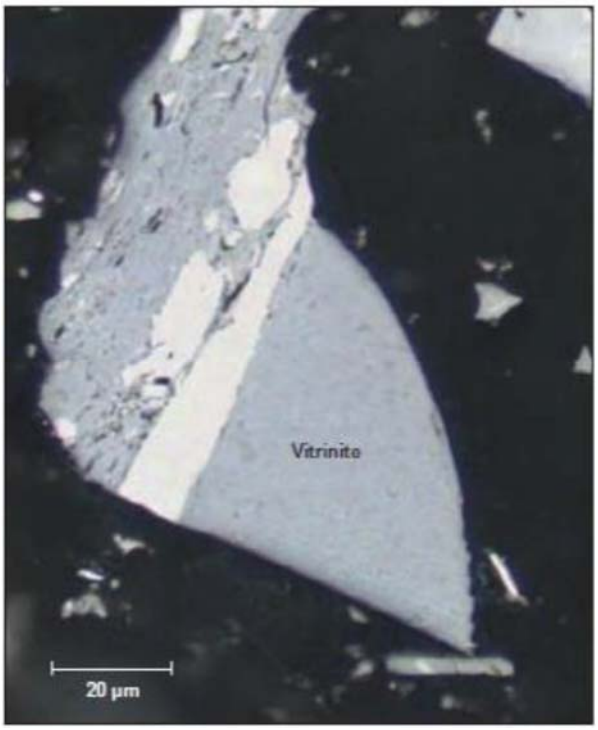
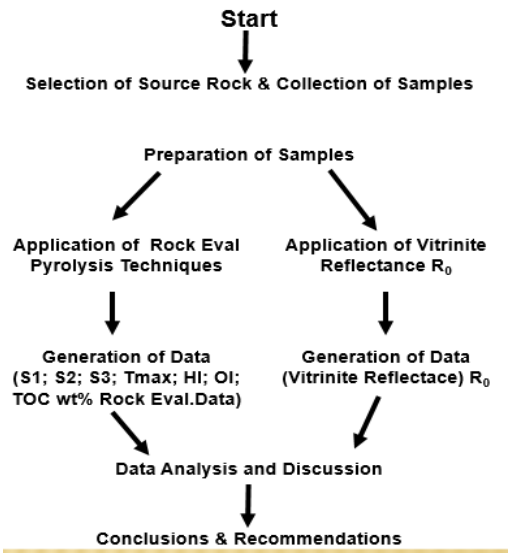


Figure-2. Virtrinite Reflectance Method (McCarthy *et al.*, 2011).

METHODOLOGY



RESULTS AND DISCUSSIONS

Figure-3 and Table-3 show an example of pyrogram obtained from TOC/Rock-Eval pyrolysis and data obtained from TOC/Rock-Eval pyrolysis and Vitrinite Reflectance method respectively.

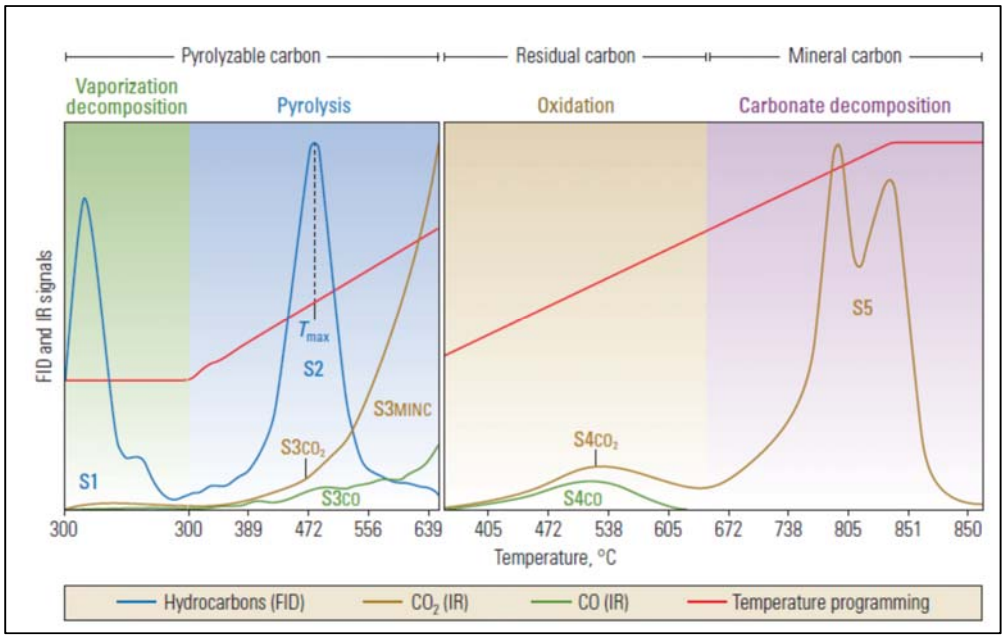


Figure-3. An example of pyrogram obtained from TOC/Rock-Eval pyrolysis(McCarthy *et al*., 2011).

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Table-3. Data obtained from TOC/Rock-Eval pyrolysis and Vitrinite Reflectance method.

Rock Eval Pyrolysis results of samples from Eocene Subathu Formation Jammu, Northwest Himalaya, India												
S No	**Type**	**S1**	**S2**	**PI**	**Tmax**	**S3**	**PC (%)**	**RC (%)**	**TOC (%)**	**HI**	**OI**	**VRo (%)**
Bergoa Coal Mine,Kalakot												
1	Coaly Shale	2.66	15.54	0.15	502	0.09	1.54	28.86	30.4	51	0	1.9
2	Coaly Shale	1.03	13.21	0.07	498	0.17	1.2	23.36	24.56	54	1	1.8
3	Carb. Shale	0.06	2.25	0.03	512	0.07	0.2	6.46	6.66	34	1	2.1
4	Carb. Shale	0.05	0.83	0.06	542	0.12	0.09	4.34	4.43	19	3	2.6
Chakkar Coal Mine, Kotla												
5	Carb. Shale	0.19	1.63	0.11	496	0.02	0.16	5.61	5.77	28	0	1.8
6	Carb. Shale	0.09	0.94	0.09	501	0.01	0.1	4.39	4.49	21	0	1.9
7	Carb. Shale	0.06	0.94	0.06	504	0	0.09	4.37	4.46	21	0	1.9
8	Carb. Shale	0.49	1.07	0.32	499	0.02	0.14	4.62	4.76	22	0	1.8
9	Carb. Shale	0.11	0.51	0.18	502	0.01	0.05	3.22	3.27	16	0	1.9
10	Carb. Shale	0.06	0.88	0.06	494	0.01	0.09	4.07	4.16	21	0	1.7
Mahogla Coal Mine, Mahogla												
11	Carb. Shale	0.13	2.26	0.05	518	0.05	0.2	6.73	6.93	33	1	2.2
12	Carb. Shale	0.05	2.51	0.02	535	0.04	0.23	10.49	10.72	23	0	2.5
13	Carb. Shale	0.21	6.57	0.03	495	0.06	0.6	13.9	14.5	45	0	1.8
14	Carb. Shale	0.33	6.5	0.05	499	0.11	0.59	10.81	11.4	57	1	1.8
15	Carb. Shale	0.05	0.63	0.07	517	0.02	0.06	2.36	2.42	26	1	2.1
16	Carb. Shale	0.12	7.72	0.02	498	0.05	0.68	13.27	13.95	55	0	1.8
17	Carb. Shale	0.15	2.5	0.06	515	0.06	0.23	7.73	7.96	31	1	2.1

A. Conceptual Framework for Source Rock Evaluation using TOC/Rock-Eval Pyrolysis and Vitrinite Reflectance Techniques

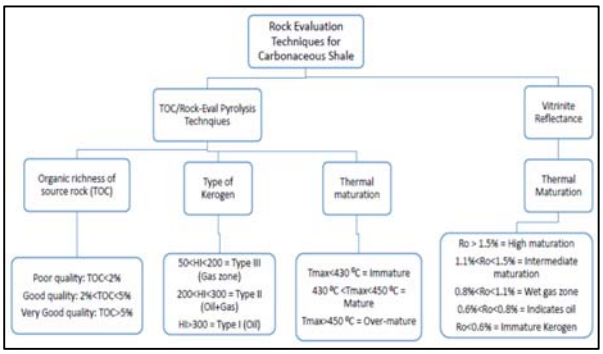

Figure-4. Conceptual Framework for Source Rock Evaluation.

B. Criteria used in Source Rock Evaluation

TOC/Rock-Eval pyrolysis techniques

Table-4. Classification of source rock based on TOC and S2 (McCarthy *et al*., 2011).

Source Rock Quality	TOC (%)	Pyrolysis S2 (mg HC/g rock)
None	<0.5	<2.0
Poor	0.5 to 1.0	2.0 to 3.0
Fair	1.0 to 2.0	3.0 to 5.0
Good	2.0 to 5.0	5.0 to 10.0
Very Good	>5.0	>10.0

Table-5. Classification of kerogen type based on hydrogen index (McCarthy *et al*., 2011).

Kerogen Type	Product Type	Hydrogen Index
I	Gas	>300
II	Gas and Oil	200 to 300
III	Oil	50 to 200

Table-6. Classification of kerogen type based on hydrogen index (McCarthy *et al*., 2011).

Maturity Level	T_{max} (°C)
Immature	<435
Mature	435 to 455
Transitional	455 to 470
Over-mature	>470

Vitrinite reflectance

Table-7. Source rock evaluation based on R_o value (McCarthy *et al*., 2011).

R_o (%)	Descriptions
>1.5	High maturation values, indicates the presence of dry gas.
1.1 to 1.5	Intermediate maturation values indicate gas with a tendency toward oil generation at the lower end of the range.
0.8 to 1.1	Wet gas can be found in this range.
0.6 to 0.8	Indicates oil
<0.6	Immature kerogen

C. Discussion on the hydrocarbon generating potential of Subathu Shales, Jammu

Organic richness

According to (Nunez-Betelu, 1994), in order to obtain reliable results from Rock-Eval pyrolysis, there must be a minimum amount of organic matter in a source rock. The outcrops obtained from Subathu formation are shales samples. By looking at the data obtained from pyrolysis as shown in Table-3 above, TOC value of Subathu formation ranged from 2.42% to 30.4%. High TOC values indicate that the shale samples are between "good" and "very good" source rock. However without sufficient burial depth, hydrocarbon will not be generated as hydrocarbon generation requires sufficient heat and pressure. Besides that, the value of S1 and S2 also showing high values. S1 which represents the amount of free hydrocarbon in the source rock ranged between 0.1 to 2.66 mg HC/g rock. S2 which represents hydrocarbon generated through pyrolysis ranged between 0.63 to 15.54 mg HC/g rock. S1, S2 and TOC values for Subathu formation showing that the shales sample has the fair to good potential to generate hydrocarbon.

Type of kerogen

Identification of the type of organic matter is essential in predicting the type of hydrocarbon to be generated from that particular source rock. Each type of kerogen is varied in terms of their origin depositional environment or burial history. These factors caused each type of kerogen to have varied hydrogen index and oxygen index. Figure-5 below shows the standard HI against OI correlation that categorizes the type of kerogen.

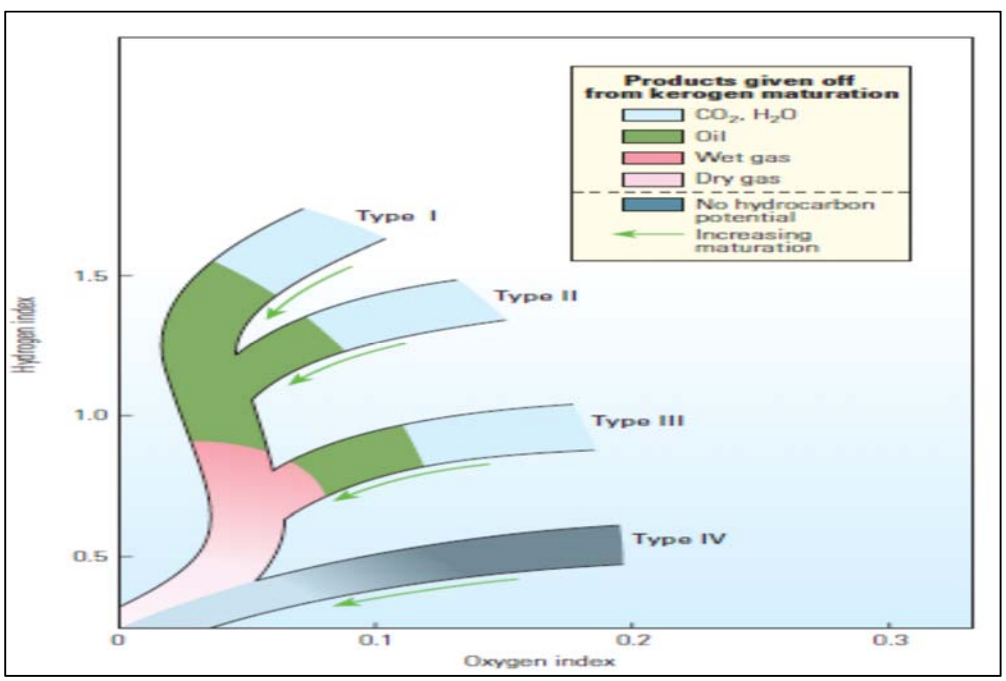

Figure-5. Standard HI versus OI correlation in determining the type of kerogen (McCarthy *et al*., 2011).

According to HI versus OI correlation as shown in Figure-6, the Subathu shales showing a trend of Type III kerogen as it contains low HI (hydrogen index) ranged below 200.

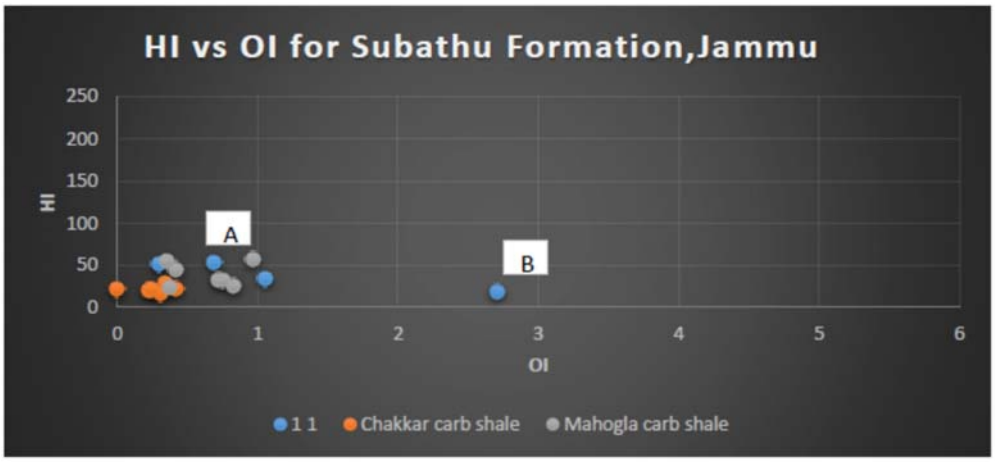

Figure-6. HI versus OI correlation for Subahtu Formation, Jammu.

Shales in Subathu formation showing a low value of OI indicating that the depositional environment is not highly oxygenated and hence there is a lower possibility for hydrogen to be a loss from source rock through oxidation. This indicates that low HI is caused by the loss of hydrogen through hydrocarbon generation before the samples were collected.

Thermal maturity

Thermal maturation is one of the most significant factors which will determine the hydrocarbon generation potential of a source rock. Thermal maturation of rocks is depending on its burial depth, pressure and time. Source rock at a different level of maturity will generate a different type of hydrocarbon. By referring to the T_{max} obtained from pyrolysis data as shown in Figure-7, T_{max} for all shale samples is higher than 470 ˚C.

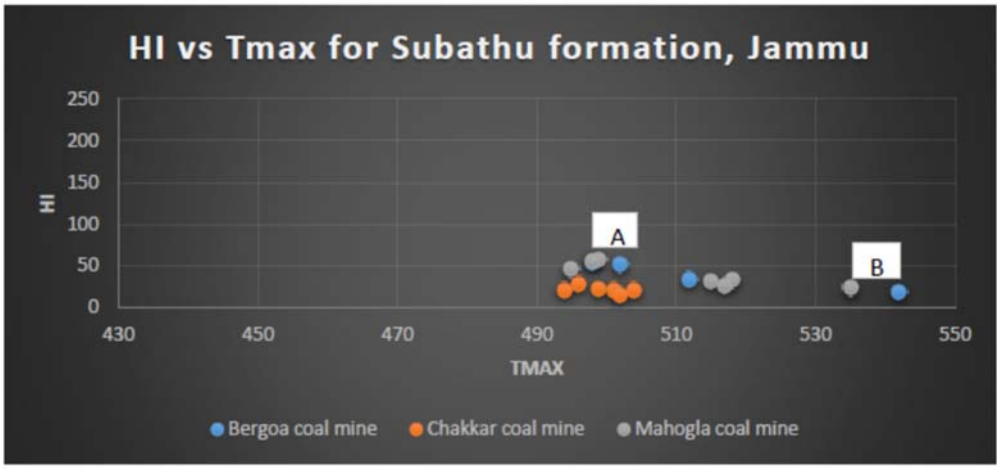

Figure-7. HI versus Tmax correlation in determining the type of kerogen.

According to the criteria listed in Table-6, it indicates that shales sample of Subathu formation are a Type III kerogen which is over-mature and it is fall at a dry gas window. Other than that, shales in Subathu formation also showing high reflectivity value (R_o) in vitrinite reflectance method with R_o higher than 1.5% indicates source rock are at high maturation stage with dry gas generation potential. Therefore, it indicates that shales in Subathu formation have fair to an excellent gas generating potential by evaluating on its TOC, S2, HI, OI, and T_{max}. Moreover, by comparing the HI versus T_{max} plot for between Subathu formation and standard HI vs T_{max} plot, it confirms that shales in Subathu formation are an over-mature Type III kerogen with gas generating potential.

CONCLUSIONS AND RECOMMENDATION

Data obtained from TOC/Rock-Eval pyrolysis and vitrinite reflectance show that the source rock of Subathu formation in Jammu region has fair to an excellent gas generating potential. This means that shales are a potential unconventional gas reservoir. T_{max} and reflectivity value (R_o) from vitrinite reflectance method also showing that the shales are at high maturation window. Then from HI versus OI plot, the graph shows that the samples collected fall at an over-mature window with a trend of Type III kerogen

which has low hydrogen index. Based on this interpretation, it poves that the shales of Subathu formation have been subjected to sufficient burial depth and thermal maturity for significant hydrocarbon generation potential.

There are some limitations in TOC/Rock-Eval pyrolysis application if the samples are critical samples. Data obtained from Rock-Eval will be erroneous when the S2 value of the rock is too low. A low S2 peak will spread widely and cause the analyzer to have difficulty in recording and determining the correct T_{max} which is used in thermal maturity interpretation. It is recommended to use other evaluation techniques such as gas chromatography, thermal alteration index or spore coloration index with Rock-Eval pyrolysis to obtain more reliable results.

Besides that, it is also recommended that Rock-Eval pyrolysis technique is applied on source rock evaluation on core sample only instead of drill cutting because the contaminants in drill cutting will cause erroneous result and it will affect the evaluation.

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