ASSESSMENT THE HYDROCARBON POTENTIALITY OF THE MIDDLE JURASSIC SARGELU FORMATION, NORTHERN IRAQ

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ABSTRACT

The Middle Jurassic Sargelu Formation is evaluated for its hydrocarbon generation potential in three wells near Duhok Governorate, Northern Iraq using ten borehole cutting samples analyzed by Rock-Eval pyrolysis, gas chromatography coupled with gas chromatography-mass spectrometry (MSS) and stable isotope. The results showed that the total organic carbon and genetic potential of the formation ranges from 0.5 to 11.6 wt. % and 1.7 to 66.3 mg HC/g of rock, respectively indicating fair to excellent source rock petroleum potential, with hydrogen index values between 270 to 555 mg HC/g TOC. The results also show kerogen Types II and mixed II/III reflecting thermal maturity within the oil generation zone. The estimated ratios of Pr/Ph, Pr/n-C₁₇ and Ph/n-C₁₈ from the extracted bitumen reveal primary accumulations of marine organic matter under reducing conditions. The $\delta^{13}C_{Sat}$ and $\delta^{13}C_{Aro}$ for the extracted bitumen range from -29.5 to -27.5‰ and -28.4 to -27.2‰, respectively. The biomarkers results indicate a significant contribution of marine organic matters preserved under relatively anoxic conditions.

Keywords: Sargelu; Rock-Eval; NW Iraq; Middle Jurassic; Biomarker

INTRODUCTION

The northern part of Iraq is characterized by multiple source rocks, one of these is rocks Sargelu Formation (Pitman *et al.*, 2004). The formation has been studied in detail for its lithology and stratigraphy by Wetzel (1948) (Bellen *et al.*, 1959).

The identification of a petroleum source rock can be established by determination of organic matter richness (quantity), type of kerogen (quality) and eventually, the maturity of source rock (Tissot and Welte, 1984, Waples, 1985 and Hunt, 1996). The Middle Jurassic deposits have been considered as potential source rocks for hydrocarbons deposited under the euxinic environment in the Middle East (Murris, 1980 and Beydoun, 1986) and in the Kurdistan Region of Northern Iraq (Al-Ameri *et al.*, 2014, English *et al.*, 2015 and Hakimi & Najaf, 2016).

Biomarkers studies are very helpful because they can provide information on the organic matter (OM) in the source like environmental conditions during its deposition and thermal maturity experienced by rock or oil (catagenesis), the degree of biodegradation along with some aspects of source rock with deposition environment, and age (Peters & Moldowan, 1993).

The possible petroleum source rocks of Jurassic and Cretaceous in Northern Iraq is still and will remain an active research area to establish the source of crude in the reservoir and their conditions of deposition. The current research is an effort in that line based on geochemical studies including Rock-Eval pyrolysis, gas chromatography (GC), stable carbon isotope and gas chromatography-mass spectrometry (GC-MSS) analysis. The main aims are to investigate total organic carbon (TOC), pyrolysis, residual oil extractions and biomarker fingerprints of the Middle Jurassic Sargelu Formation to provide details on the origin of OM, thermal maturity and depositional environment.

GEOLOGICAL SETTING

This study is the first one conducted on the Middle Jurassic Sargelu Formation in three wells in northwestern Iraq; Mangesh-1 (Ma-1), Atrush-1 (At-1) and Shaikhan-8 (Sh-8) wells (Fig. 1). Iraqi Kurdistan Region forms the NE boundary of the Arabian Plate (AP) and is a part of the Alpine Mountain Belt. This belt has an E–W trend in the northern part and NW–SE trend in the northeastern part (Jassim and Buday, 2006). The study area is a part of the Zagros Basin and representing the so-called Zagros Fold Belt of Northern Iraq (Ameen, 1992). Zagros basin is a narrow belt that extends from northern Iraq to the Arabian Gulf. Two main sectors are identified within the Taurus-Zagros Belt; the Thrust Zone and the Folded Zone (Ameen, 1992) (Fig. 2). The Folded Zone has been subdivided into two zones based on folding intensity (Jassim and Goff, 2006). They are (1) the High Folded Zone, which consists of a series of NW–SE trending, mostly asymmetrical anticlines separated by narrower synclines characterized by steeper southern or southwestern limbs; and (2) the Foothill Zone which is characterized by low altitude hills and relatively smaller anticlines and synclines where the prominent Hemrin-Mekhlul mountain chain

forms its southwestern boundary, separating it from the Mesopotamian Zone. Atrush and Shaikhan oilfields are located in the High Folded Zone, a part of the Zagros Fold and Thrust Belt of Iraq, some 85 km northwest and 60 km north of Erbil city respectively. Both are recent oil discoveries in the Kurdistan Region of Iraq. The Atrush structure is a NW–SE oriented, doubly-plunged asymmetrical anticline, and its northeastern limb is gentler than the southwestern one (Al-Naqib, 1980).

Fig. 1: Landsat image showing the location of the studied wells, Ma-1, At-1 and Sh-8

Fig. 2: Tectonic Map of Northern Iraq (redrawn after Buday, 1980 and Ameen, 1992)

The Shaikhan anticline is a WNW–ESE trending doubly-plunged asymmetrical anticline, with a gentle southwestern limb and steeper back limb. This fold is considered as an open structure due to its interlimb angle of 120° (Al-Azzawi and Hamdoon, 2008). During the Jurassic period, all Northern Iraq lands were considered parts of an intrashelf compartment that was geometrically separated from the basin physiography (Jassim and Goff, 2006) and many formations were deposited in the intrashelf and basins. The first description of the Sargelu Formation was introduced by Wetzel (1948) from an outcrop of about 115 m thick at Surdash Anticline, Sulaimaniya area within the High Folded Zone of Iraq (Bellen *et al.*, 1959).

The Middle Jurassic Sargelu Formation generally crops out at some limbs and cores of structures in the Highly Folded Zones of Northern Iraq (Buday, 1980). This formation represents the basal supersequence (Upper Aalenian-Bathonian) of the Megasequence AP7 which includes Alan, Sargelu, Naokelekan and Barsarin formations.

The stratigraphic successions exposed within the studied well-sections include Jurassic Alan, Sargelu, Naokelekan and Barsarin formations from the oldest to youngest. Younger Cretaceous units are comprised of Chia Gara, Garagu, Sarmord, Qamchuqa, Kometan, Wajna, and Aqra formations. These were further overlain by the Tertiary succession comprised of Kolosh, Gercus and Pila Spi formations.

The outcrops of Sargelu Formation in several localities such as Banik, Gara, and Naokelekan consist of thinly bedded black bituminous limestone, interbedded with dolomitic limestone and black papery shale intercalated by thin black, streaks of chert nodules in the uppermost part of the formation (Balaky, 2004). This formation in Iraq was probably deposited under sulphate-reducing marine conditions (Murriss, 1980; Al-Ameri and Zumberge, 2012; Al-Ameri *et al.*, 2014).

The lithologic description of the Sargelu Formation is based on the subsurface sections in this study (Fig. 3). The Mangesh-1 well (Ma-1) section shows different lithologies such as calcareous dolomite, dolomitic limestone and limestone. In Atrush-1 well (At-1), the lithology is dominated by a combination of fine-grained limestone, and light grey to dark grey shale. The limestone fraction contains calcite and some anhydrite. However, in well Shaikhan-8 (Sh-8), the formation consists of fine-grained limestone, argillaceous limestone interbedded with thin layers of anhydrite and limestone intercalated with marl and shale (Fig. 3).

Fig. 3: Stratigraphic Columns of Sargelu Formation in Wells Ma-1, At-1 and Sh-8

METHODOLOGY

This study is mainly based on the analyses of cutting samples collected from shales, argillaceous limestone, and dolomite fraction selected from wells in the northwestern part of Iraqi Kurdistan Region (Fig. 3). The samples were picked from different stratigraphic levels, representing various depth intervals. There are ten cutting samples of the Sargelu Formation, it is from Wells Ma-1 (from depth 2545 to 2647 m), At-1 (from depth 1212 to 1245 m), and Sh-8 (from depth 1570 to 1666 m). They were collected to pyrolysis analysis. These cuttings were stored at the Geological Survey Repository in Erbil City.

The utilized analytical techniques were pyrolysis and biomarker. Four samples were analyzed by GC, and three samples were used to stable carbon isotope and GC-MSS analysis. These analyses were performed by Strato Chem Laboratories, Cairo, Egypt, in collaboration with GeoMark Research, Ltd. in Houston, Texas, USA.

Pyrolysis

The Rock-Eval pyrolysis data provides information on the quality, quantity and maturity level of OM of source rocks (Tissot and Welte, 1984 and Peters, 1986). Primarily, the samples were cleaned from contaminants from drilling mud additives by washing them with the distal water several times until no mud was visible on their surface. For the Rock-Eval Pyrolysis, a small amount of sample (100 mg) is heated in an inert atmosphere (helium or nitrogen) then combusted with air to acquire several key geochemical parameters related to the hydrocarbon potential of the rock such as TOC (Peters, 1986). TOC (wt.%) is calculated as the sum of organic carbon and residual organic carbon.

Pyrolysis provides four geochemical parameters, including S_1 , which represents free and adsorbed hydrocarbons in mg HC/g of rock, S_2 , which refers to generated hydrocarbons in mg HC/g of rock, S_3 , which provides CO_2 released during the pyrolysis phase of the analysis in mg CO_2 /g of rock, and T_{max} , which gives the temperature of maximum pyrolytic hydrocarbon yield in °C. These primary parameters are used to deduce the values of TOC (wt.%). The provided parameters can be used to differentiate the kerogen types and oil migration by using the following equations:

$$\text{Oxygen Index (OI)} = (S_3/\text{TOC}) \times 100, \text{ in mg } CO_2/\text{g TOC} \quad (1)$$

$$\text{Hydrogen Index (HI)} = (S_2/\text{TOC}) \times 100, \text{ in mg HC/g TOC} \quad (2)$$

$$\text{Production Index (PI)} = S_1/(S_1+S_2) \quad (3)$$

$$\text{Genetic Potential (GP)} = S_1+S_2, \text{ in kg HC/ton of rock} \quad (4)$$

Gas Chromatography and Stable Carbon Isotope

The GC analysis of saturated hydrocarbon fractions in the extracted bitumens was carried out using an Agilent 7890 chromatograph equipped with a flame ionization detector (FID). The oven temperature program included an initial rise from 70 to 300 °C at a rate of 3 °C/min, then held for 30 min at 300 °C. Gas chromatography-mass spectroscopy (GC-MSS) analysis was accomplished for the saturated and aromatic fractions in the source rock extracts with an Agilent 7890A gas chromatograph coupled to an Agilent 5975C mass spectrometer. The gas chromatograph oven was initially run at 60 °C for 2 min, programmed to 300 °C at 2 °C/min and held at 300 °C for 30 min. Bulk stable carbon isotope analysis was implemented for the saturated and aromatic hydrocarbon fractions by an online system utilizing a Finnigan delta plus mass spectrometer coupled with a Carlo Erba EA 1108 instrument.

RESULTS AND DISCUSSION

The analysis results of TOC% and Rock-Eval pyrolysis are given in Table 1, while those of stable carbon isotope composition, GC, and biomarker are shown in Table (2).

Table 1: Rock-Eval Pyrolysis and TOC results of Sargelu Formation in the studied wells

Well	Depth (m)	TOC Wt%	S ₁	S ₂	S ₃	T _{max} °C	HI	OI	PI	GP	S ₁ /TOC	R _o %
Ma-1	2607–2610	0.51	0.34	1.37	1.04	429	270	205	0.	1.71	0.67	0.
	2628–2631	0.62	0.50	2.01	0.86	430	325	139	0.	2.51	0.81	0.
	2646–2649	0.70	0.44	2.28	0.89	429	326	127	0.	2.72	0.63	0.
At-1	1215–1220	3.06	1.38	14.0	0.54	436	459	18	0.	15.4	0.45	0.
	1235–1240	8.59	2.80	47.7	0.71	438	555	8	0.	50.5	0.33	0.
	1245–1250	5.53	2.06	28.9	0.63	435	524	11	0.	31.0	0.37	0.
Sh-8	1608–1611	1.16	0.49	4.97	0.82	422	428	71	0.	5.46	0.42	0.
	1626–1629	4.16	1.89	18.8	0.72	436	454	17	0.	20.7	0.45	0.
	1656–1659	11.60	2.52	63.7	0.90	440	550	8	0.	66.2	0.22	0.
	1661–1664	4.48	1.43	23.9	0.72	436	535	16	0.	25.3	0.32	0.

TOC = Total Organic Carbon, wt%; S₁ = Free hydrocarbon content, mg HC/g rock; S₂: Remaining hydrocarbon generative potential, mg HC/g rock; S₃ = Carbon dioxide yield, mg CO₂/g rock; T_{max} = Temperature at maximum of S₂ peak; HI: Hydrogen Index = S₂×100/TOC, mg HC/g TOC; OI: Oxygen Index = S₃×100/TOC, mg CO₂/g TOC; PI: Production Index = S₁/(S₁+S₂); GP: Genetic Potential = (S₁+S₂), Kg HC/Ton rock; R_o= Calculated Vitrinite Reflectance.

Table 2: Gas Chromatograph-Mass Spectrometry coupled with Isotope data

Well	Depth (m)	Sample weight	δ ¹³ C _{Sat.} (‰) ^(a)	δ ¹³ C _{Aro.} (‰) ^(b)	CV	Pr/ph	Pr/n-C ₁₇	Ph/n-C ₁₈	CPI	C ₂₇ % Sterane	C ₂₈ % Sterane	C ₂₉ % Sterane
Ma-1	2631	75.70	-27.50	-27.20	-2.47	0.66	0.24	0.28	0.86	55.90	16.00	28.10
At-1	1240	90.00	-29.50	-28.40	-2.28	0.63	0.21	0.42	0.94	54.20	15.50	30.30
Sh-8	1629	62.40				0.58	0.34	0.53	0.94			
	1664	96.10	-28.20	-28.10	-2.68	0.62	0.32	0.46	0.93	53.20	14.70	32.10

^(a)δ¹³C_{Sat.} = Stable Carbon isotopic composition of the Saturated HC fraction

^(b)δ¹³C_{Aro.} = Stable Carbon isotopic composition of the Aromatic HC fraction

Organic Matter Richness and Petroleum Potential

The TOC% versus S₁ plot of Hunt (1996), applied to the studied samples of Sargelu Formation, shows that they are not contaminated and of indigenous origin (Fig. 4a). The TOC% content of the sediments can be considered as a direct expression of kerogen and bitumen abundance (Hunt, 1996). Sediments with TOC% values of 0.5 to 1.0 wt.% are considered as potential source rocks (Tissot and Welte, 1984; Tissot *et al.*, 1987; Hunt, 1996). The result show that TOC% ranges from 0.51 to 11.6 wt.% (average = 4.04 wt.%) (Table 1).

Fig. 4: (a) TOC% versus S₁ for identifying migrated hydrocarbons for Sargelu Fn. (b) Pyrolysis S₂ versus TOC% plot showing generative source rock potential (c) HI versus Pyrolysis T_{max} for the analyzed samples (d) Plot of Pyrolysis S₂ versus TOC% shows the kerogen type of the studied samples. The plot also indicates whether kerogen is oil or gas prone

Most of the samples have fair to excellent genetic potential (GP) based on the classification given by Peters and Cassa (1994) (Fig. 4b), and the GP contents range from 1.71 to 66.27 kg HC/ton of rock (average = 22.17 kg HC/ton of rock). Samples of Ma-1 well have poor GP, averaging 2.47 kg HC/ton of rock. On the other hand, samples of At-1 and Sh-8 wells have excellent GP, with an average 32.32 kg HC/ton of rock and 29.47 kg HC /ton of rock, respectively (Table 1). The hydrocarbon yields (S₂) are in

agreement with TOC% content in At-1 and Sh-8 wells, indicating that the Sargelu sediments are excellent source rock on the contrary of Ma-1 well (Fig. 4b).

Quality of Organic Matter

Type II kerogen generates oil, while Type III kerogen produces gas, if the source rock was buried deeply enough (Hunt, 1996). The HI versus $T_{\max}$ plot of Van Krevelen applied on the studied samples indicated the presence of kerogen Type II for At-1 and Sh-8 wells with mixed kerogen Types II and III for Ma-1 well (Fig. 4c). Most of the samples are placed within oil window field; however, two samples from Ma-1 well and one from Sh-8 well behaved differently as they were placed in the areas of early maturation stage and immature stage, respectively (Fig. 4c), owing to their low $T_{\max}$ values ($< 430^{\circ}\text{C}$) (Table 1).

The results of the analysis show that the HI ranges from 270 to 555 mg HC/g TOC. Most HI values of Sargelu Formation samples are > 300 mg HC/g TOC, illustrating Type II kerogen source with except one sample from Ma-1 well, that has a HI < 300 mg HC/g TOC, which is more likely as a mixture of Types II and III kerogen.

Rock-Eval pyrolysis $T_{\max}$ values of the Sargelu Formation samples range from 422 to 440 $^{\circ}\text{C}$. Such $T_{\max}$ values are considered as low for Type I kerogen, high for Type II and much higher for Type III kerogen. The reason for such variations is due to the structural complexities of OM (Tissot and Welte, 1984, Peters, 1986 and Bordenave *et al.*, 1993).

Rock-Eval Pyrolysis suggests that variations among the results are considerable because of the limestone- and dolostone-dominated rocks for Sargelu Formation. Carbonate rocks commonly affect the OI, thus lowering the identification of kerogen types within the formation (Abdula, 2015). The high values of OI in Ma-1 well samples (Table 1) indicate possible oxidation has taken place during the generation of HC, creating oxic conditions in the depositional environment.

The S_2 versus TOC plot shows that most of Sargelu Formation samples tend to be Type II Kerogen with different levels of oil and gas prone (Fig. 4d). In general, the results show different kerogen types, with a tendency toward Type II in the majority of the Sargelu Formation samples (Figs. 4a, b, c and d).

The calculated Vitrinite Reflectance (R_o) values, obtained by converting $T_{\max}$ (Peters *et al.*, 2005).

$$R_o (\text{calculated}) = (0.018 \times T_{\max}) - 7.16 \quad (5)$$

In order to get the real value of R_o , the above formula was used for samples that have S_2 values > 0.5 mg HC/g rock and $T_{\max} > 420$ or $< 500^{\circ}\text{C}$.

The expelled oil from source rocks starts when the ratio of S_1 to TOC ranges between 0.1 and 0.2 (Hunt, 1996). All S_1 /TOC values of Sargelu Formation are > 0.2 (Table 1). Therefore, the expulsion of oil from Sargelu Formation occurred at depths shallower than where it exists; 2607 m, 1215 m, and 1608 m in Ma-1, At-1, and Sh-8 wells, respectively.

Normal Alkanes and Isoprenoids

The Pr/Ph ratio is frequently used as an indicator of the depositional environment and maturity detection (Peters *et al.*, 2005). When this ratio is (< 1), this would be used as a proxy to indicate reducing or anoxic conditions of carbonate source rocks, mainly when followed by the high porphyrin and sulfur contents (Peters and Moldowan, 1993). The lower pristane (Pr) content within a sample is mainly attributed to reducing the environment, however, the higher Pr/Ph ratio has likely caused oxygenic environment at the deposition time of source rock (Ten Haven *et al.*, 1987).

The organic-rich anoxic carbonate rocks generally generate oil with Pr/Ph ratio of < 2 (Peters *et al.*, 2005). The ratios of the Pr/Ph of the four extracts are low, ranging between 0.58 to 0.66, indicating anoxic, reduced marine carbonate depositional environment (Fig. 5).

Fig. 5: Gas Chromatographs of extracted rock samples from Sargelu Formation, NW Iraq

All extract samples show large unresolved complex mixture (UCM) or hump, where the n-alkanes and isoprenoids still survive and remain unaltered. These samples appear to have suffered from excessive evaporation where Pr and Ph still retain their characters.

The Pr/n-C₁₇ (0.21 to 0.34) and Ph/n-C₁₈ (0.28 to 0.53) ratios (Table 2) suggest primary accumulation of marine organic matter under reducing conditions instead of maturity effect or biodegradation for extracts (Fig. 6).

Fig. 6: Pr/n-C₁₇ versus Ph/n-C₁₈ of the Sargelu Formation samples extract for the studied wells

The carbon preference index (CPI) can be used as a maturity indicator (Peters *et al.*, 2005).

$$CPI = 2(C_{23} + C_{25} + C_{27} + C_{29}) / [C_{22} + 2(C_{24} + C_{26} + C_{28}) + C_{30}] \dots (6)$$

For instance, immature source rocks have CPI values > 1.2 or < 0.8 (Moldowan *et al.*, 1985). CPI values below 1.0 are rare and indicate extracts formed in hypersaline or carbonate environments (Peters *et al.*, 2005). The calculated CPI of the extracts of Sargelu Formation were < 1.2 and > 0.8 (Table 2), indicating marine carbonate environment and maturation stage.

Sable Carbon Isotopes Composition ($\delta^{13}C_{\text{‰}}$)

The isotope composition of the extracted bitumen from saturates and aromatics fraction can be used to distinguish between oxic terrigenous and an anoxic marine environment by applying a mathematical relation known as canonical variable (CV), (Sofer, 1984).

$$CV = -2.53\delta^{13}C_{Sat} + 2.22\delta^{13}C_{Aro} - 11.65 \quad (7)$$

Values of $CV > 0.47$ indicate predominant terrigenous organic matter, while $CV < 0.47$ indicates mostly marine organic source. The calculated CV of the studied extracts ranges from -2.68 to -2.28 (Table 2), which confirms the prevailing marine environment.

Additionally, carbon isotope values of saturates ($\delta^{13}C_{Sat}$) and aromatics ($\delta^{13}C_{Aro}$) for three rock extract samples within Sargelu Formation range from -27.5 to -29.5‰ and -27.2 to -28.4‰, respectively (Table 2). These values display a slight variation in the isotopic composition of the extracted samples (Demaison and Huizinga, 1991) and confirm that the OM within the studied formation was mainly derived from the marine origin (Fig. 7).

Fig. 7: Isotope $\delta^{13}C$ from Saturate versus $\delta^{13}C$ Aromatic cross plot for the analyzed studied samples extract

The ternary diagram of $C_{27}\%$ - $C_{28}\%$ - $C_{29}\%$ steranes compounds from the extracts of source rocks were frequently used to identify the type or origin of the initial OM. The main sources of C_{27} steranes are of marine origin, and that of C_{29} steranes are mostly derived from advanced plants. The C_{28} steranes consist of a mixture of advanced plants and mainly algae (Peters *et al.*, 2005). The examined samples, including rock-extract of Sargelu Formation, are characterized by the abundance of C_{27} over C_{28} and C_{29} (Fig. 8), which proves deposition was done in the marine environment (Fig. 9).

Fig. 8: Mass Chromatograms of Steranes, m/z 217, from the extract of the studied sample

Fig. 9: Steranes Ternary Diagram of C₂₇%, C₂₈% and C₂₉% of the selected extract samples using GC-MSS m/z 217

CONCLUSIONS

The following conclusions could be drawn from the results of organic geochemistry and biomarker studies of the Middle Jurassic Sargelu Formation samples taken from three exploration wells drilled in NW Iraqi Kurdistan .

- (1) The OM of Sargelu Formation was kerogen Types II and mixed II/III, that the kerogen thermally mature and frequently located within the oil generation zone.
- (2) The studied samples were characterized by marine OM dominated with high TOC% contents.
- (3) The isoprenoids ratio of Pr/Ph range was between (0.58 to 0.66), Pr/n-C₁₇ (0.21 to 0.34) and Ph/n-C₁₈ (0.28 to 0.53), of the extracts, are low, indicating mature with anoxic, reducing marine carbonate depositional origin.
- (4) Stable isotopic ratios of the saturate and aromatic fractions, expressed as a Ternary plot between regular C₂₇% - C₂₈% - C₂₉%, steranes, revealed that the existing OM is mainly derived from the marine depositional environment.

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