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Petroleum Geochemistry of the Varicolored Shale of the Nubian Formation in Wells R8-82, R9-82, and R10-82, Sirte Basin, North Central Libya

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الجيوكيمياء البترولية للصخر الزيتي متعدد الألوان التابع لتكوين النوبة في الآبار *R8-82* و *R9-82* و*R10-82*، حوض سرت، شمال وسط ليبيا

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Abstract:
A mineralogical and geochemical evaluation of the Varicolored Shale of the Nubian Formation (Early Cretaceous) was carried out in wells R8-82, R9-82, and R10-82, Sirte Basin, north central Libya. Regarding mineral composition, the studied shale in well R8-82 mainly consists of clay minerals with smaller amounts of quartz, carbonates (calcite and dolomite), and potassium feldspar. The shale is classified into three lithofacies: (1) Argillaceous shale (CM); (2) Silica rich-argillaceous shale (CM-1); and (3) Mixed argillaceous shale (CM-2). Regarding source rock evaluation, there are four categories of organic richness in the shale: poor, fair, good, and very good. The shale is dominated by hydrogen-poor kerogens (II–III, III, and IV). The degree of thermal maturity varies from immature to late mature. The shale mainly contains indigenous hydrocarbons with lesser amount of nonindigenous hydrocarbons. The shale is principally a source of oil. The shale is deemed inappropriate for the extraction of shale oil. Regarding shale oil evaluation, mature oil predominates in the shale. There are two oil families; the first family is derived from carbonate or evaporite, while the shale or clay-rich rock is the source of the second family. The majority of these families are marine in origin.

Keywords: Mineral Composition, Petroleum Geochemistry, Shale Oil, Varicolored Shale, Nubian Formation, Sirte Basin, Libya.

الملخص
أُجري تقييم معدني وجيوكيميائي لطفلة فاريوكولورد التابعة لتكوين النوبة (العصر الطباشيري المبكر) في الآبار R8-82 و R9-82وR10-82، في حوض سرت، شمال وسط ليبيا. فيما يتعلق بالتركيب المعدني، تتكون الطفلة المدروسة في البئر R8-82 بشكل رئيسي من معادن طينية مع كميات أقل من الكوارتز والكربونات (الكالسيت والدولوميت) والفلسبار البوتاسي. صُنفت الطفلة إلى ثلاث وحدات صخرية: (1) طفلة طينية(CM) ؛ (2) طفلة طينية غنية بالسيليكا(CM-1) ؛ و(3) طفلة طينية مختلطة .(CM-2) أما

فيما يخص تقييم الصخور المصدرية، فتوجد أربع فئات من حيث غنى الطفلة بالمواد العضوية: فقيرة، متوسطة، جيدة، وجيدة جدًا. تهيمن على الطفلة الكيروجينات الفقيرة بالهيدروجين(II-III ، III، و IV). وتتراوح درجة النضج الحراري من غير ناضجة إلى ناضجة تمامًا. يحتوي الصخر الزيتي بشكل رئيسي على هيدروكربونات محلية مع كمية أقل من الهيدروكربونات غير المحلية. يُعد الصخر الزيتي مصدرًا أساسيًا للنفط، إلا أنه يُعتبر غير مناسب لاستخراج النفط الصخري. عند تقييم النفط الصخري، نجد أن النفط الناضج هو السائد فيه. توجد مجموعتان من النفط؛ الأولى مشتقة من الكربونات أو المتبخرات، بينما تُعد الصخور الطينية أو الصخر الزيتي مصدرًا للمجموعة الثانية. معظم هاتين المجموعتين ذوات أصل بحري.

الكلمات المفتاحية: التركيب المعدني، جيوكيمياء البترول، النفط الصخري، الصخر الزيتي متعدد الألوان، تكوين النوبة، حوض سرت، ليبيا.

1. Introduction

The Sirte Basin is a major sedimentary basin in central Libya, located south of the Gulf of Sirte. The basin covers an area of approximately 600,000 km^2. It lies mostly beneath gravel plains and sand seas, with little surface expression. The basin contains up to 7,500 m of sediments. It is Libya's youngest, richest, and largest sedimentary basin, containing the country's largest petroleum reserves and ranking 13th globally (Hallett and Clark-Lowes, 2016). Fig. 1 displays the stratigraphic chart of the Sirte Basin. The basin is a prolific hydrocarbon province with a Meso-Cenozoic petroleum system, rich in oil and gas fields (e.g., Ahlbrandt, 2001; Burwood *et al.*, 2003; Saheel *et al.*, 2010; Abdunaser, 2015; Albriki *et al.*, 2021, 2024). Its origin is attributed to the collapse of the Sirte Arch during the Late Jurassic to Early Cretaceous (Thomas, 1995; Hallett, 2002; Saleem, 2015). The basin has a complex history of tectonic activity (Abdunaser and McCaffrey, 2014).

Desio (1935) coined the term Nubian Sandstone in Libya. The word "Nubian" is not included in the geological map of Libya. Petroleum geologists in Libya still frequently use the term Nubian Sandstone, especially in the southeast Sirte Basin (Barr and Weegar, 1972). In the eastern Sirte Basin, however, the terms Sarir Sandstone and Kalanshiyu Sandstone are used instead of the Nubian Sandstone (Hallett, 2002). Ibrahim (1991) considered that the typical section of the Nubian Formation is found in well R3-82 (thickness is about 1143 m). The formation in the typical section is divided into three units: (1) The Lower Nubian Sandstone; (2) The Variegated Shale; and (3) The Upper Nubian Sandstone. Ambrose (2000) showed that the Sarir Formation can be categorized into five units: (1) The Lower Sarir Sandstone; (2) The Red Shale; (3) The Middle Sarir Sandstone; (4) The Variegated Shale; and (5) The Upper Sarir Sandstone. The Variegated Shale is an important source rock, the Lower Nubian Sandstone (Middle Sarir Sandstone) and the Upper Nubian Sandstone (Upper Sarir Sandstone) are primary reservoirs in the Sirte Basin (Ibrahim, 1991; Ambrose, 2000). The Nubian Formation consists of fluvial and lacustrine sediments. It is primarily of Early Cretaceous age (Hallett, 2002). The Agip Oil Company adopted the classification of Ibrahim (1991) for the Nubian Formation, but used the term Varicolored Shale instead of Variegated Shale.

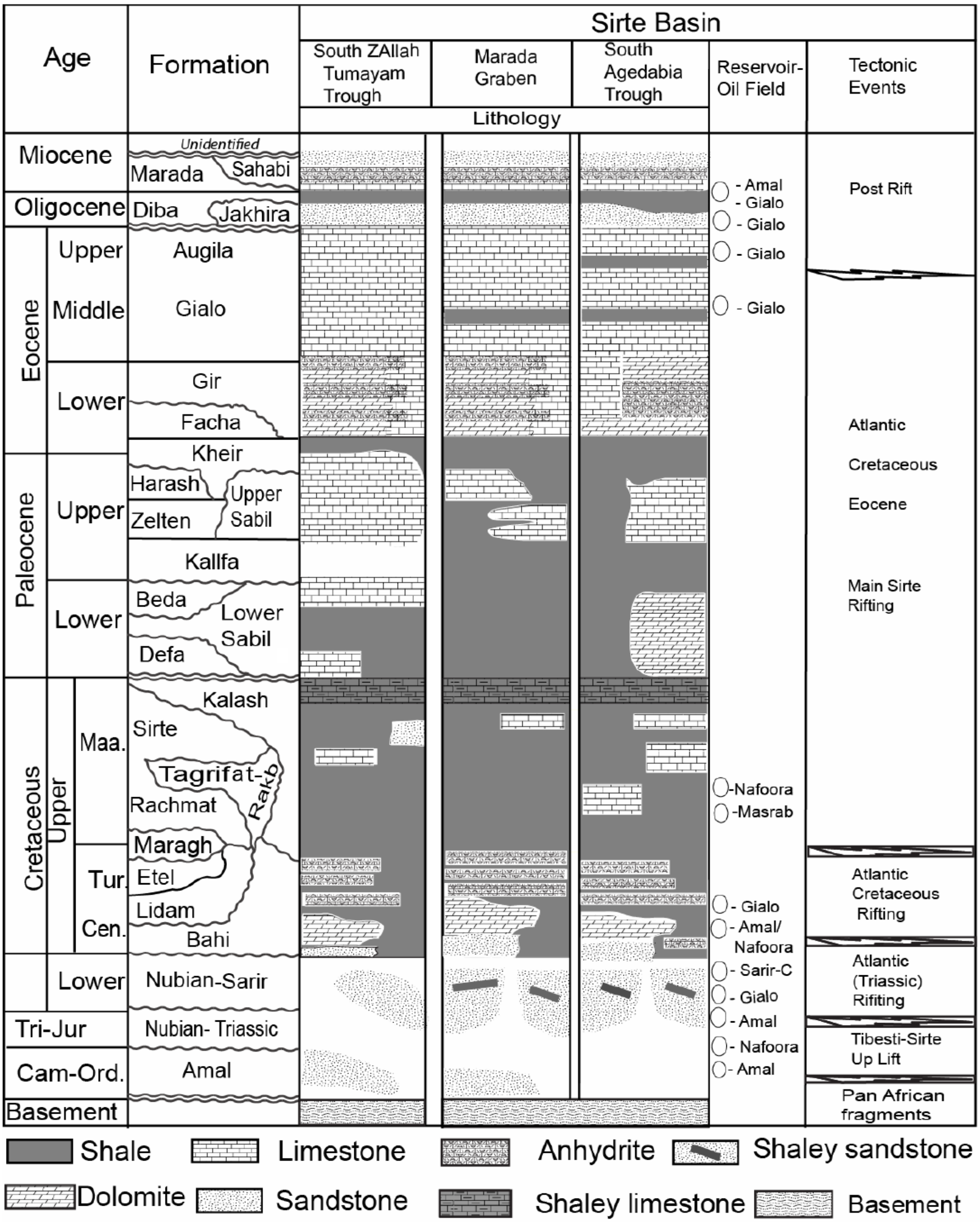

Fig. 1: Stratigraphic column of the Sirte Basin (drawn by Barr and Weeger, 1972 and modified by Aboglila et al., 2010).

Assessing the mineral composition and petroleum geochemistry of the Varicolored Shale of the Nubian Formation in wells R8-82, R9-82, and R10-82, Sirte Basin, north central Libya (Fig. 2), is the purpose of this work. The studied wells are located in the eastern part of the Sirte Basin. According to the earlier studies (Burwood *et al.*, 2003; Aboglila and Elkhalgi, 2013; USGS, 2019; Albriki *et al.,* 2021), there is no published work of the Varicolored Shale in wells R8-82, R9-82, and R10-82.

2. Methodology

This work was based on the mineral and chemical data obtained from the Mellitah Oil and Gas B.V.. Below is the work plan:

(1) A total of 32 cutting shale samples were selected (11 samples from well R8-82 at 12270 to 13857.2 ft, 17 samples from well R9-82 at 11990 to 13500 ft, and 4 samples from well R10-82 at 14906 to 14933.5 ft). Moreover, 4 oil samples were extracted from the shale samples (two samples from well R8-82 at 13855.1 and 13857.2 ft, one sample from well R9-82 at 13487 ft, and one sample from well R10-82 at 14933.5 ft).

(2) X-ray diffraction (XRD) examination was used to assess the mineral composition of seven samples from well R8-82 (at depths of 13670 to 13857.2 ft).

(3) Rock-Eval pyrolysis was utilized to ascertain the source rock characteristics of the shale samples (except for samples at a depth of 13855.1 and 13857.2 ft in well R8-82).

(4) Utilizing gas chromatography (GC), the biomarker data of the oil samples were acquired.

(5) The carbon isotope values of the saturated, aromatic, resin, and asphaltene fractions were determined using isotope ratio mass spectrometry (IRMS).

The abbreviations and equations for the parameters utilized in this study are shown in Table 1. Table 2 displays the interpretation of the parameters.

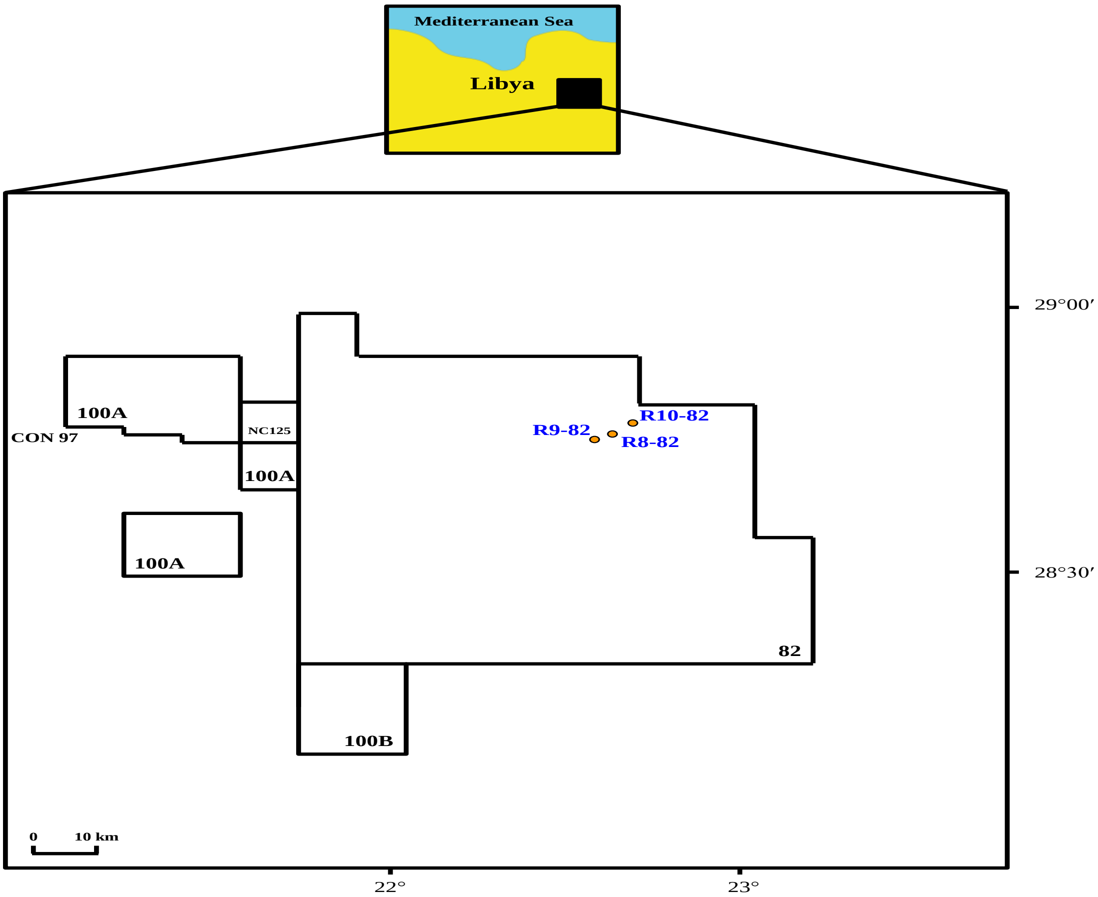

Fig. 2: Composite map showing the location of the studied wells.

3. Results and Discussion

3.1. Mineral Composition

The mineral composition of seven samples of the Varicolored Shale from well R8-82 was determined (Table 3). It is clear that potassium feldspar is only noticeable in the sample at a depth of 13855.1 ft. Furthermore, carbonate minerals are missing in the samples at depths of 13855.1 and 13857.2 ft. The shale of the Varicolored Shale in well R8-82 belongs to the argillaceous category (Fig. 3). There are three lithofacies: CM, CM-1, and CM-2 (argillaceous shale, silica rich-argillaceous shale, and mixed argillaceous shale, respectively).

3.2. Source Rock Evaluation

To evaluate the source rock, Rock-Eval pyrolysis was performed on thirty samples from wells R8-82, R9-82, and R10-82 (Table 4). The studied shale showed four levels of organic richness (poor, fair, good, and very good, Figs. 4 and 5). Hydrogen-poor kerogens (II-III, III, and IV) predominated in the Varicolored Shale (Figs. 6 and 7). The thermal maturity levels range from immature to late mature (Figs. 8 and 9). Except for samples at depths of 11990 to 12470 ft in well R9-82, all of the examined samples exhibit S1/T values less than 1.5, demonstrating the predominance of indigenous hydrocarbons in the Varicolored Shale (Fig. 10). The shale is primarily a source of oil (Fig. 11). The OSI is less than 100 in the studied samples, except for four samples at depths of 11990 to 12470 ft in well R9-82 (Fig. 12). Therefore, the Varicolored Shale in the studied wells is considered unsuitable for shale oil extraction.

Table 1: Abbreviations and equations of the parameters used in this work

Parameter	Abbreviation and equation
Total organic carbon (wt%)	TOC
Quantity of free hydrocarbons (mg/g)	S1
Quantity of generated hydrocarbons (mg/g)	S2
Quantity of CO_2 generated during pyrolysis (mg/g)	S3
Temperature at which maximum rate of generation of hydrocarbons occurs (°C)	T_{max}
Hydrogen index (mg/g)	HI = S2/TOC × 100
Oxygen index (mg/g)	OI = S3/TOC × 100
Oil saturation index (mg/g)	OSI = S1/TOC × 100
Production index	PI = S1/(S1+S2)
Pyrolyzable carbon index (mg/g)	PCI = 0.83(S1+S2)
Saturated hydrocarbons	SAT
Aromatic hydrocarbons	ARO
Resins plus asphaltenes	NSO
Saturated hydrocarbons, aromatic hydrocarbons and resins plus asphaltenes	SARA
Stable carbon isotope of saturated hydrocarbons (‰)	$\delta^{13}C_{SAT}$
Stable carbon isotope of aromatic hydrocarbons (‰)	$\delta^{13}C_{ARO}$
Stable carbon isotope of resins plus asphaltenes (‰)	$\delta^{13}C_{NSO}$
Vitrinite reflectance	$R_0 = 0.018 \times T_{max} - 7.16$
Argillaceous shale	CM
Silica rich-argillaceous shale	CM-1
Mixed argillaceous shale	CM-2
Carbonate rich-argillaceous shale	CM-3
Mixed shale	M
Calcareous/siliceous mixed shale	M-1
Argillaceous/siliceous mixed shale	M-2
Argillaceous/calcareous mixed shale	M-3
Calcareous shale	C
Silica rich-calcareous shale	C-1
Mixed calcareous shale	C-2
Clay rich-calcareous shale	C-3
Siliceous shale	S
Carbonate rich-siliceous shale	S-1
Mixed siliceous shale	S-2
Clay rich-siliceous shale	S-3

Table 2: Interpretation of the parameters

Objective	Parameter	Values	Interpretation	Reference
Organic richness	TOC (%)	0–0.5	Poor	Peters and Cassa (1994)
		0.5–1	Fair	
		1–2	Good	
		2–4	Very good	
		>4	Excellent	
	S1	0–0.5	Poor	
		0.5–1	Fair	
		1–2	Good	
		2–4	Very good	
		>4	Excellent	
	S2	0–2.5	Poor	
		2.5–5	Fair	
		5–10	Good	
		10–20	Very good	
		>20	Excellent	
Kerogen type	HI (mg/g)	<50	Type IV	
		50–200	Type III	
		200–300	Type II-III	
		300–600	Type II	
		>600	Type I	
	S2/S3	<1	Type IV	
		1–5	Type III	
		5–10	Type II-III	
		10–15	Type II	
		>15	Type I	
	PCI (mg/g)	<15	Type III	Reed and Ewan (1986)
		40–50	Type II	
		≥75	Type I	
Thermal maturity	T_{max} (^{o}C)	<435	Immature	Peters and Cassa (1994)
		435–445	Early mature	
		445–450	Peak mature	
		450–470	Late mature	
		>470	Postmature	
	R_0	0.2–0.6	Immature	
		0.6–0.65	Early mature	
		0.65–0.9	Peak mature	
		0.9–1.35	Late mature	
		>1.35	Postmature	
	PI	<0.1	Immature	
		0.1–0.15	Early mature	
		0.25–0.4	Peak mature	
		>0.4	Late mature	
		-	Postmature	
Indigeneity	S1/TOC	<1.5	Indigenous hydrocarbons	Hunt (1996)
		>1.5	Nonindigenous hydrocarbons	
Unconventional hydrocarbons	OSI	<100	Unsuitable potential	Tian *et al.*, (2024)
		>100	Suitable potential	
oil-source rock correlation	C_{29}/C_{30}	<1	Shale or clay-rich sources	Andrew *et al.*, (2001)
		>1	Carbonate or evaporite sources	

Table 3: Descriptive statistics of mineral composition of the Varicolored Shale in well R8-82

Minerals	N	Minimum	Maximum	Mean	Std. Deviation
Quartz	7	14	40	20.86	9.51
Potassium feldspar	7	0	2	0.29	0.76
Calcite	7	0	13	5.71	4.99
Dolomite	7	0	3	2.00	1.41
Clay minerals	7	60	79	71.14	6.04

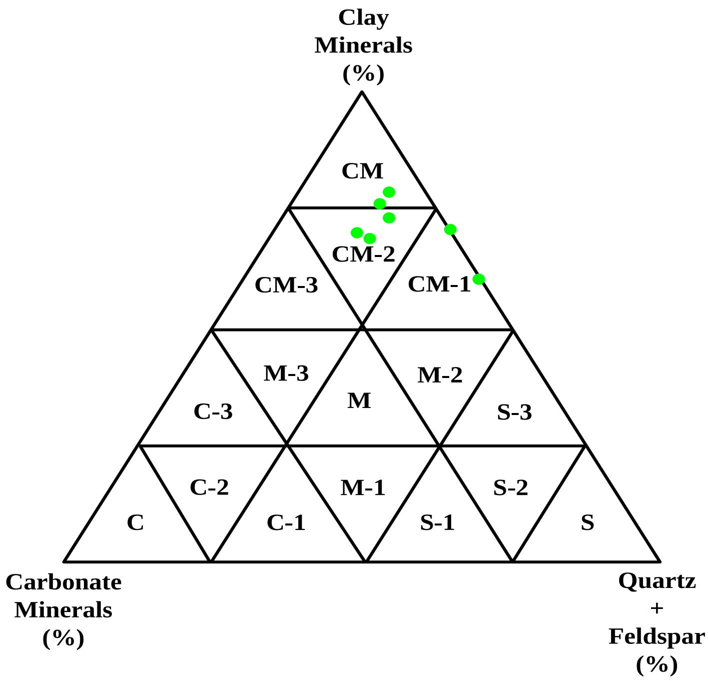

Fig. 3: Ternary plot of clay minerals-carbonate minerals-quartz+feldspar showing the classification of the Varicolored Shale in well R8-82 (fields after Gamero-Diaz et al., 2012).

Table 4: Descriptive statistics of Rock Eval pyrolysis data of the Varicolored Shale in wells R8-82, R9-82, and R10-82

Parameters	N	Minimum	Maximum	Mean	Std. Deviation
TOC	30	0.28	3.29	0.73	0.61
S1	30	0.01	3.12	0.48	0.89
S2	30	0.07	11.15	1.43	2.28
S3	30	0.16	1.41	0.57	0.33
OI	30	5	470	117.47	105.29
HI	30	15	339	145.57	86.48
T_{max}	30	427	452	440.17	6.36
R_0	30	0.53	0.98	0.76	0.11
PI	30	0.01	0.97	0.21	0.29
OSI	30	2.78	693.33	81.05	171.22
PCI	30	0.17	10.04	1.58	2

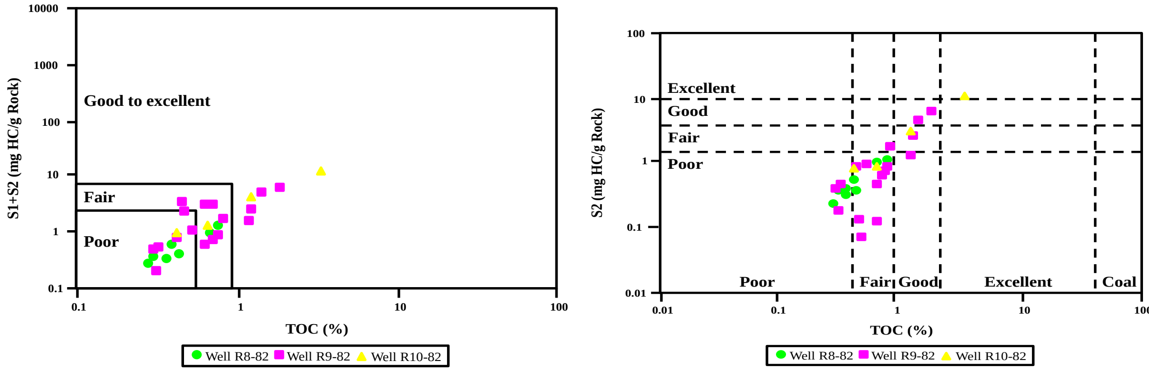

Fig. 4: Binary plots (TOC vs. S1+S2 (fields after Tissot and Welte, 1984) and TOC vs. S2 (fields after Dembicki, 2009)) showing the organic richness of the Varicolored Shale in wells R8-82, R9-82, and R10-82.

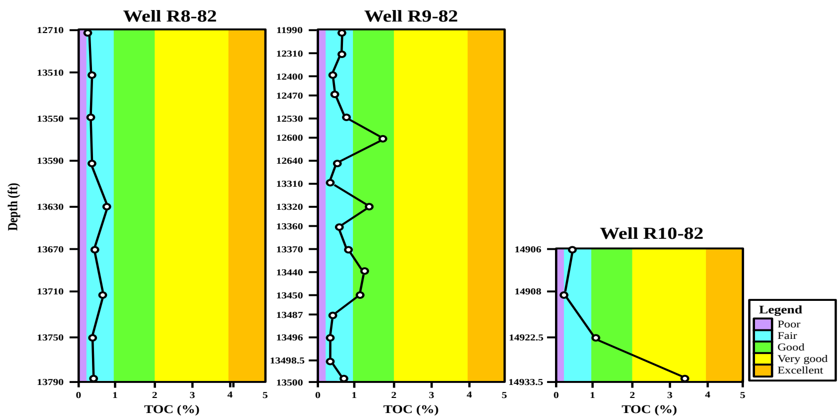

Fig. 5: Binary plots of TOC vs. depth showing the organic richness of the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Peters and Cassa, 1994).

3.3. Shale Oil Evaluation

The GC data of biomarkers of shale oil in the Varicolored Shale are shown in Table 5. Moreover, Table 6 displays the IRMS data of isotopes. It should be noted that the categorization of shale oil has not been evaluated due to the lack of the American Petroleum Institute (API) data. The Varicolored Shale appears to contain mature oil, according to the SARA plot (Fig. 13). Furthermore, the C_{29}/C_{30} values indicate that the shale oil origin in well R8-82 is carbonate or evaporite (C_{29}/C_{30} ranges from 1 to 1.18), while shale or clay-rich rock is the likely origin of the shale oil in well R9-82 (C_{29}/C_{30} = 0.79). It should be noted that the C_{29}/C_{30} ratio in well R10-82 has not been determined. In addition, the studied oil is primarily of marine origin (Fig. 14).

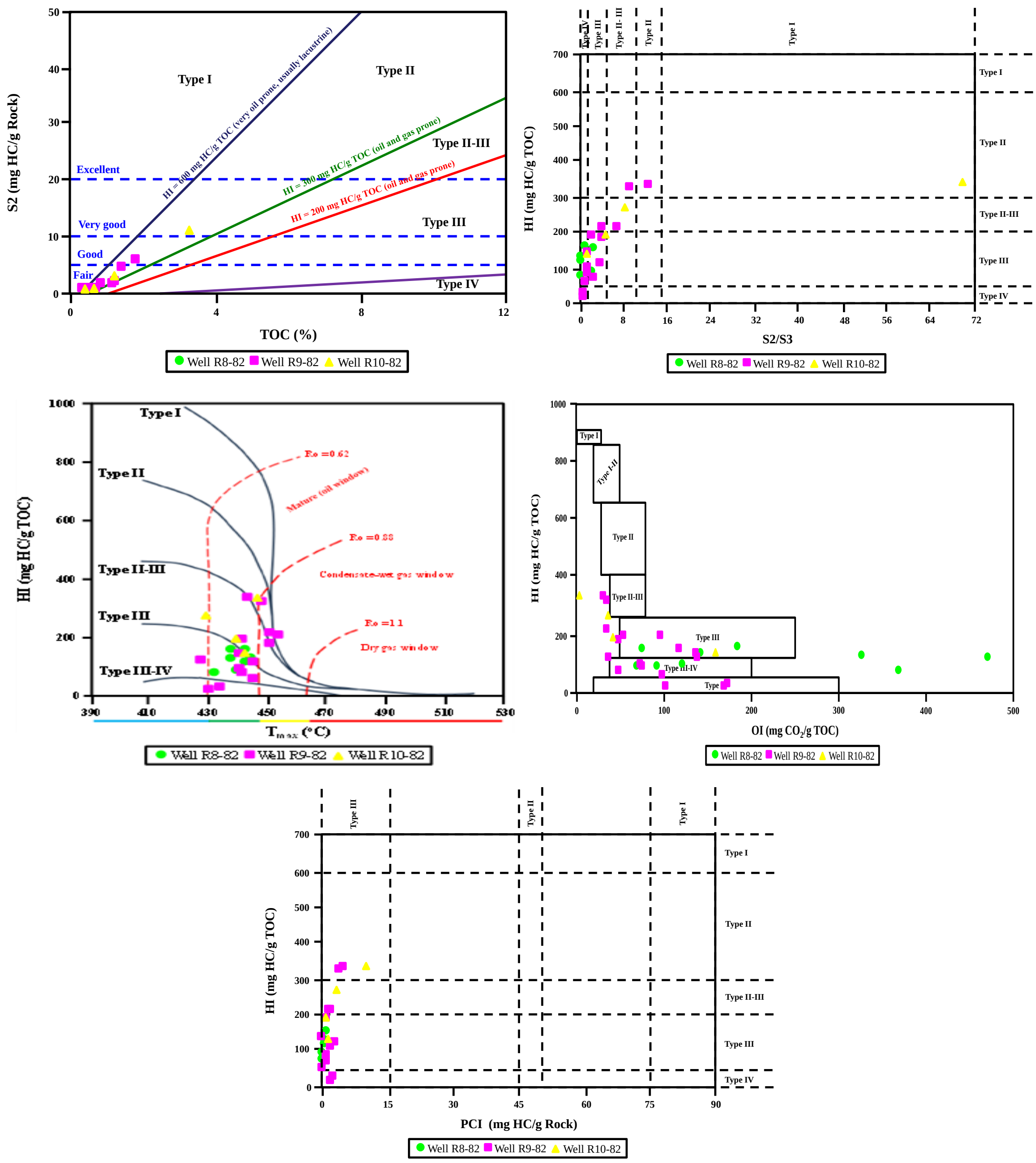

Fig. 6: Binary plots (TOC vs. S2 (fields after Longford and Blanc-Valleron, 1990), S2/S3 vs. HI (fields after Peters and Cassa, 1994), T_{max} vs. HI (fields after Hall et al., 2016), OI vs. HI (fields after Jones, 1987), and PCI vs. HI (fields after Reed and Ewan, 1986; Peters and Cassa, 1994)) showing the kerogen type of the Varicolored Shale in wells R8-82, R9-82, and R10-82.

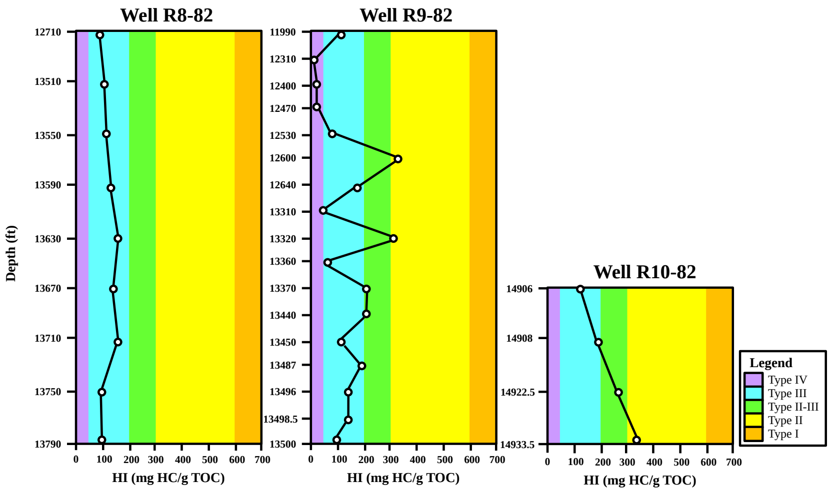

Fig. 7: Binary plots of HI vs. depth showing the kerogen type of the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Peters and Cassa, 1994).

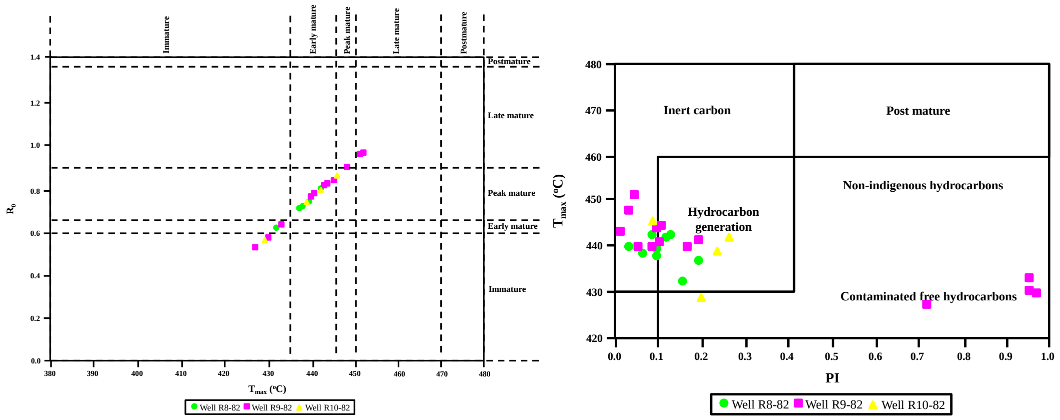

Fig. 8: Binary plots (T_{max} vs. R_0 (fields after Peters and Cassa, 1994) and PI vs. T_{max} (fields after Tissot and Welte, 1984)) showing the thermal maturity of organic matter in the Varicolored Shale in wells R8-82, R9-82, and R10-82.

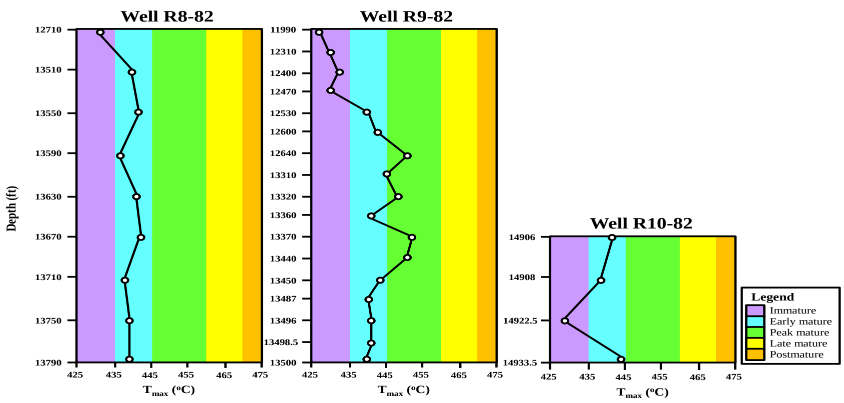

Fig. 9: Binary plots of T_{max} vs. depth showing the thermal maturity of organic matter in the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Peters and Cassa, 1994).

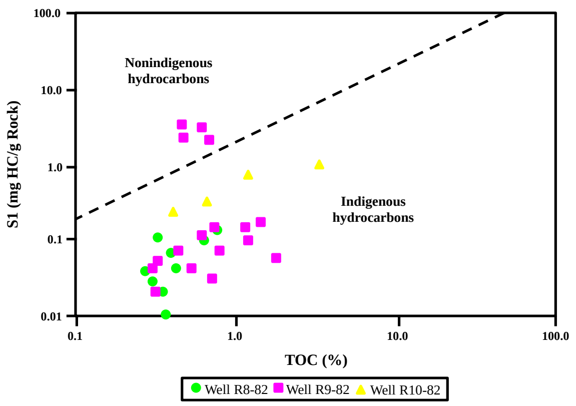

Fig. 10: Binary plot of TOC vs. S1 showing the indigeneity of hydrocarbons in the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Hunt, 1996).

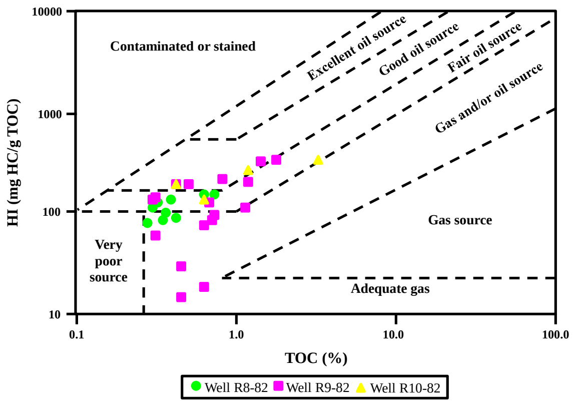

Fig. 11: Binary plot of TOC vs. HI showing the hydrocarbon potential of the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Jackson et al., 1985).

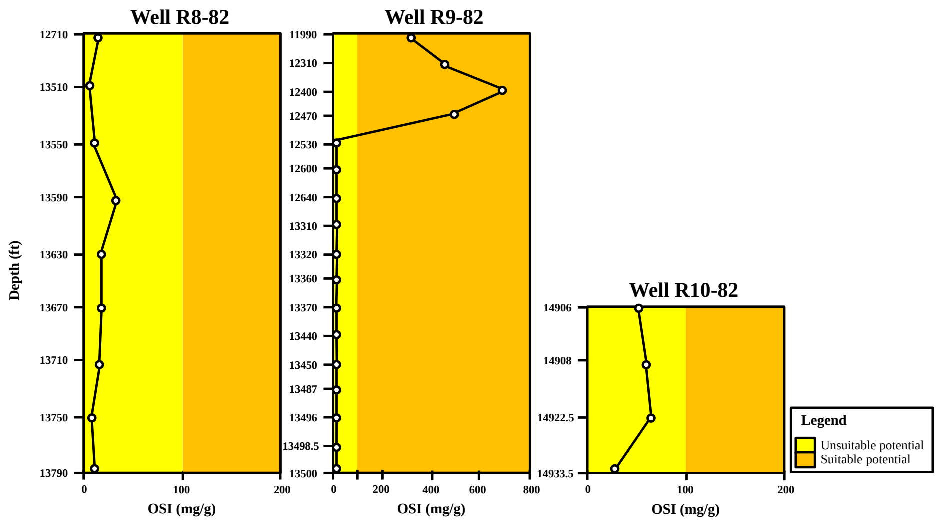

Fig. 12: Binary plots of OSI vs. depth showing the potential for recoverable shale oil from the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Tian et al., 2024).

Table 5: Descriptive statistics of GC data of shale oil in the Varicolored Shale in wells R8-82, R9-82, and R10-82

Parameters	N	Minimum	Maximum	Mean	Std. Deviation
SAT	4	50	83	62.25	14.36
ARO	4	5	21	14.25	6.7
NSO	4	12	35	23.5	9.47
C_{29}/C_{30}	3	0.79	1.18	0.99	0.2

Table 6: Descriptive statistics of IRMS data of shale oil in the Varicolored Shale in wells R8-82, R9-82, and R10-82

Parameters	N	Minimum	Maximum	Mean	Std. Deviation
$\delta^{13}C_{SAT}$	4	-32.28	-22.61	-28.49	4.18
$\delta^{13}C_{ARO}$	4	-30.62	-21.06	-28.02	4.65
$\delta^{13}C_{NSO}$	4	-30.56	-21.47	-27.93	4.32

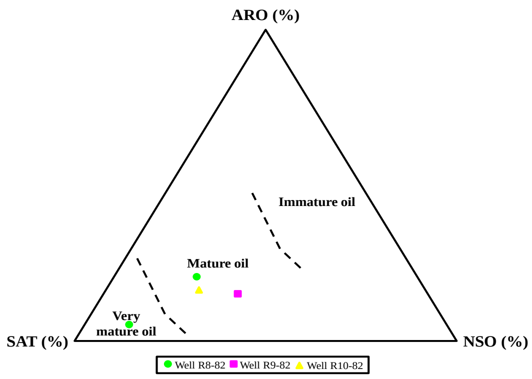

Fig. 13: Ternary plot of SARA showing the shale oil maturity in the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Peters et al., 2005).

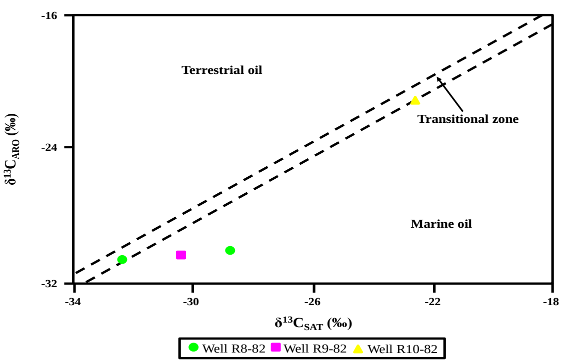

Fig. 14: Binary plot of $\delta^{13}C_{SAT}$ vs. $\delta^{13}C_{ARO}$ showing the shale oil origin in the Varicolored Shale in wells R8-82, R9-82, and R10-82 (fields after Sofer, 1984).

4. Conclusions

The Varicolored Shale of the Nubian Formation (Early Cretaceous) in wells R8-82, R9-82, and R10-82, Sirte Basin, north central Libya, was evaluated mineralogically and geochemically. Here are the conclusions:

(1) The shale in well R8-82 is primarily composed of clay minerals, with smaller concentrations of quartz, calcite, dolomite, and potassium feldspar.

(2) The shale is of the argillaceous type.

(3) The shale revealed poor to very good levels of organic richness.

(4) Kerogens of type II–III, III, and IV predominated in the shale.

(5) Immature, early mature, peak mature, and late mature were the four stages of thermal maturity that the shale displayed.

(6) Indigenous hydrocarbons make up the majority of the shale, but nonindigenous hydrocarbons are present in smaller amounts.

(7) The shale is fundamentally an oil source.

(8) The shale is thought to be unsuitable for shale oil extraction.

(9) The shale oil is of the marine type. It is characterized by maturity.

(10) Carbonate (or evaporite) and shale (or clay-rich rock) are the sources of the shale oil.

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