



# Source rock characterization of the upper Barremian, Albian and Cenomanian–Turonian organic-rich strata outcropping in Oued Bazina area, NE of Thibar diapir: Northern Tunisia

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## Abstract

Rock-Eval pyrolysis, total organic carbon (TOC) determination and gas chromatography-mass spectrometry (GC-MS) analysis were performed on samples from outcrop sections of the upper Barremian (M'Cherga Formation), Albian (Fahdene Formation) and Cenomanian–Turonian (Bahloul Formation) in the Oued Bazina area located in the NE of the diapiric structure of Thibar, northern Tunisia. The upper Barremian and Cenomanian–Turonian organic-rich strata display high TOC values with a mean of 3.39% and 2.82%, respectively, while the Albian succession deposits exhibit TOC values lesser than 2%. The half-graben structures developed at these epochs have acted as restricted mini-basins favouring the water stagnation, the accumulation and the preservation of the organic matter and consequently the development of locally euxinic conditions in the bottom waters. The generative petroleum potential ( $S_1 + S_2$ ) of the upper Barremian and Cenomanian–Turonian studied organic-rich strata is good to very good and appears to be moderate for the Albian organic-rich deposits. The n-alkane distributions of the Cretaceous source rocks are typical for a marine planktonic origin. The Pristane/Phytane ratio indicates a sub-oxic depositional environment for the upper Barremian and Albian successions and a sub-oxic to anoxic environment for the Cenomanian–Turonian organic-rich strata. Regarding the maturity degree of the three studied organic-rich strata, only the upper Barremian source rock presents a high maturity level with  $T_{max}$  values ranging from 441 °C to 448 °C, which indicates that the deposits have been deeply buried and consequently have generated hydrocarbons that have been recognized in the studied oil seep. The integration of our results with available data of  $T_{max}$  values on other outcrops in the salt dome zone allowed drafting a maturity trend of the studied source rocks. A general northeast–southwest trend of maturity increase is observed with  $T_{max}$  values varying from 436 to 446 °C.

**Keywords** Northern Tunisia · Organic geochemistry · Cretaceous source rocks · Oued Bazina · Thibar salt dome

## Introduction

The Cretaceous period (upper Barremian, Albian and Cenomanian–Turonian time intervals) (Fig. 1a) is manifested by a greenhouse climate state as a consequence of the manifestation of large igneous provinces (Barron and Washington 1985; Arthur et al. 1985; Leckie et al. 2002; Bodin et al. 2015). It is also characterized by a decrease in the tectonic activity and registered high sea level (e.g. Jenkyns 1980; Skelton et al. 2003). The high temperature recorded during this period and the acceleration of the hydrological cycle (Menegatti et al. 1998) have increased the primary productivity and led to euxinic conditions (Föllmi et al. 1994; Föllmi 2012) with the occurrence of distinctive episodes of organic-rich deposit accumulations called oceanic anoxic events (OAEs; Jenkyns 1980). The Cretaceous oceanic anoxic events are well identified and

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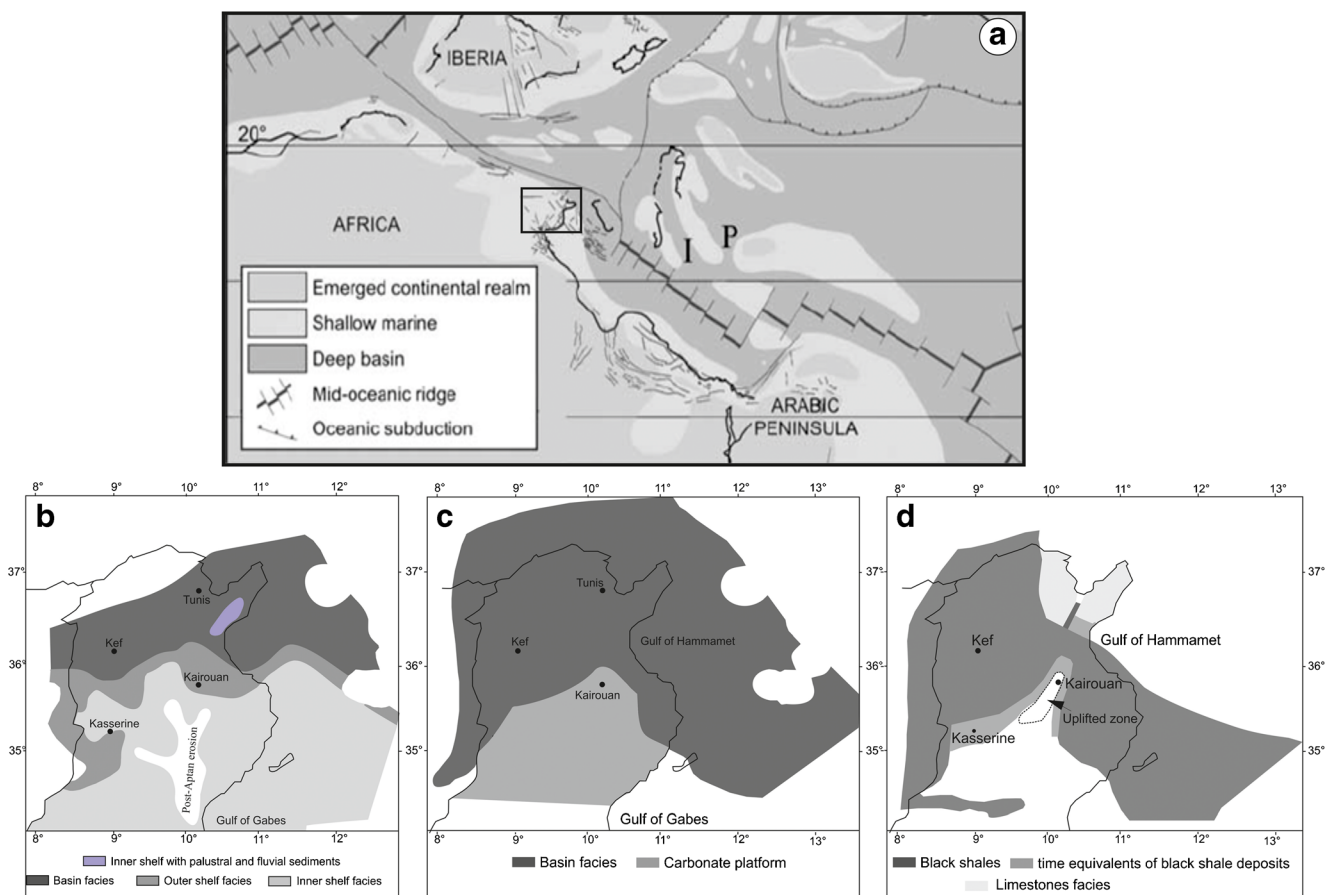
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**Fig. 1** **a** Aptian–Cenomanian paleogeography of western Tethys (Dercourt et al. 1993). **b** Facies distribution during the early Aptian age (modified from Zghal and Arnaud-Vanneau 2005). **c** Facies distribution

during the Albian age (Chihouei et al. 2010). **d** Facies distribution during the Cenomanian–Turonian age (modified from Zagrani et al. 2008; Soua et al. 2009)

studied in the northern Tethys Margin (Jenkyns 1980; Cecca and Pallini 1994; Leckie et al. 2002; Jenkyns 2010). The OAE1a event known as “Selli Level” was of the first identified and the most widely studied one (Arthur et al. 1990; Baudin et al. 1998). It is represented by 1 to 3 m thick prominent regional black shales in the Umbria–Marche Basin where total organic carbon (TOC) values achieve 18% (Coccioni et al. 1987). OAE1b event is marked by the deposition of laminated organic-rich strata that were recognized in several locations in France, Germany and Austria. Also, this event (OAE1b) was recognized later across several regions of the Tethyan–Atlantic domains (Leckie et al. 2002). OAE1d has been recorded in the Umbria–Marche Basin and is composed of black shale layers interbedded with limestones exhibiting TOC values more than 5%. The OAE2 is widespread across and outside the Tethyan margin. It corresponds to *Actinocamax plenus* marls from the south of England (Jefferies 1963; Gale et al. 1993; Paul et al. 1999); to the “Thomel Level” of the Vocontian basin (Crumiere et al. 1990; Grosheny et al. 2006); and to the “Bonarelli Level” in central Italy (Arthur and Premoli-Silva 1982). In northern Tunisia, the OAE1a event occurs among the succession of

M’Cherga Formation (Fig. 1b), while the OAE1b, c and d events of the Albian period are represented by the Fahdene Formation (Fig. 1c), and finally, the OAE2 event of Cenomanian–Turonian transition age (Fig. 1d) corresponds to the Bahloul Formation (Memmi 1989; Ben Ferjani et al. 1990; Layeb 1990; Saidi 1993; Talbi 1993; Lüning et al. 2004; Zagrani et al. 2008; Elkhazri et al. 2009; Heldt et al. 2010; Soua 2010, 2011; Ben Fadhel et al. 2011; Affouri et al. 2013; Layeb et al. 2013; Ben Fadhel et al. 2014; Soua 2016; Touati 2017). The Bahloul Formation represents the main Cretaceous source rock in onshore and offshore Tunisia and records high TOC values reaching 10.45% (Layeb 1990; Soua 2011; Affouri et al. 2013; Soua 2013, 2016; Ayari 2015; Touati 2017). In northern Tunisia, the Albian oceanic anoxic events OAE1b and OAE1c present TOC values ranging between 0.17 and 3.43% and contain mixed type II/III organic matter (Saidi 1993; Khalifa et al. 2018; Hallek 2019; Hallek et al. 2020). On the contrary to the OAEs 1b, c, d and 2, the early Aptian OAE1a event is marked by low to moderate TOC values (Elkhazri et al. 2009; Talbi et al. 2019). It is worth noting that a few stratigraphic and geochemical researches have been

carried out on the upper Barremian level which is considered as a potential source rock in Tunisia, and some recent biostratigraphic works consider it as of Bedoulian age (Elkhazri et al. 2009).

We emphasize that in the NE of the Thibar salt dome area, few bulk geochemical studies (Layeb 1990; Affouri et al. 2013; Touati 2017; Khalifa et al. 2018) have interested the Cretaceous oceanic anoxic sub-events comparatively with their analogues in central Tunisia (Soua 2016) and in the Dorsal domain (Elkhazri et al. 2009; Soua 2016; Talbi et al. 2019). In this contribution, we aim to (1) study and evaluate the organic-rich deposits of the upper Barremian to the lower Turonian time intervals; (2) to determine the genetic link between the Cretaceous source rocks and the oil seep recorded in the NE of the Thibar salt dome structure; and (3) to better understand the depositional conditions and the maturity evolution by studying the vertical distribution of the upper Barremian, Albian and Cenomanian–Turonian organic-rich facies. The bulk geochemical characterization of the main Cretaceous source rocks and the oil seep/ source rock correlation may help to reconsider the petroleum system of the study area. Furthermore, our data will be integrated with previous studies in order to make a comparison with other localities situated in the salt dome zone. This comparison is of major interest to understand the role played by local factors (i.e. tilted block basin architecture and Triassic halokinetic activities) in the installation of anoxic conditions and the thermal maturity variation.

## Geological and stratigraphic setting

During the Cretaceous time, an extensional tectonic regime prevailed in the main sedimentary basins of the southern Tethyan margin. Such extensional tectonic experienced a strong phase during the Jurassic to the Hauterivian, followed, during the Barremian, by an interval of moderate tectonic movements (Rabhi and Ben Ayed 1990) but with larger subsidence rates, which favoured the development of wide and thick shales and carbonate rocks. In northern Tunisia, it is thought that the basin was segmented into NE–SW tilted blocks that controlled the organic-rich strata deposition (Chaari 2002; Melki et al. 2012; Belayouni et al. 2013). At least four major NE–SW master faults have controlled the sedimentation and the architecture of the basin (Fig. 2a) corresponding to Cap–Serrat–Ghardilaou fault (CSGF), Ras Korane–Thibar fault (RKTF), El Alia–Teborsouk fault (ETF), Tunis–Ellès Fault (TEF) and Zaghouan fault (ZF) (Zargouni 1978; Burollet and Ellouz 1986; Martinez et al. 1991; Bouaziz et al. 2002; Melki et al. 2012; Belayouni et al. 2013). These major faults controlled differential subsidence and sedimentation patterns at least during Barremian–Albian epochs and have been reactivated later for several

times until the late Eocene and during the Miocene Alpine phase (e.g. Perthuisot 1978; Rabhi and Ben Ayed 1990; Masrouhi et al. 2008; Khomsi et al. 2009). The Cretaceous deposits studied here are involved in the half anticline of Oued Bazina which lies in the NE of the Thibar salt dome structure (Fig. 2b). Nevertheless, the studied outcrop sections appear to be slightly different to those of the salt dome succession and show more affinity to deposits of the “Medjerda valley zone”.

## Lithological succession of the studied sections

### Upper Barremian organic-rich strata of Douar Mraoua section

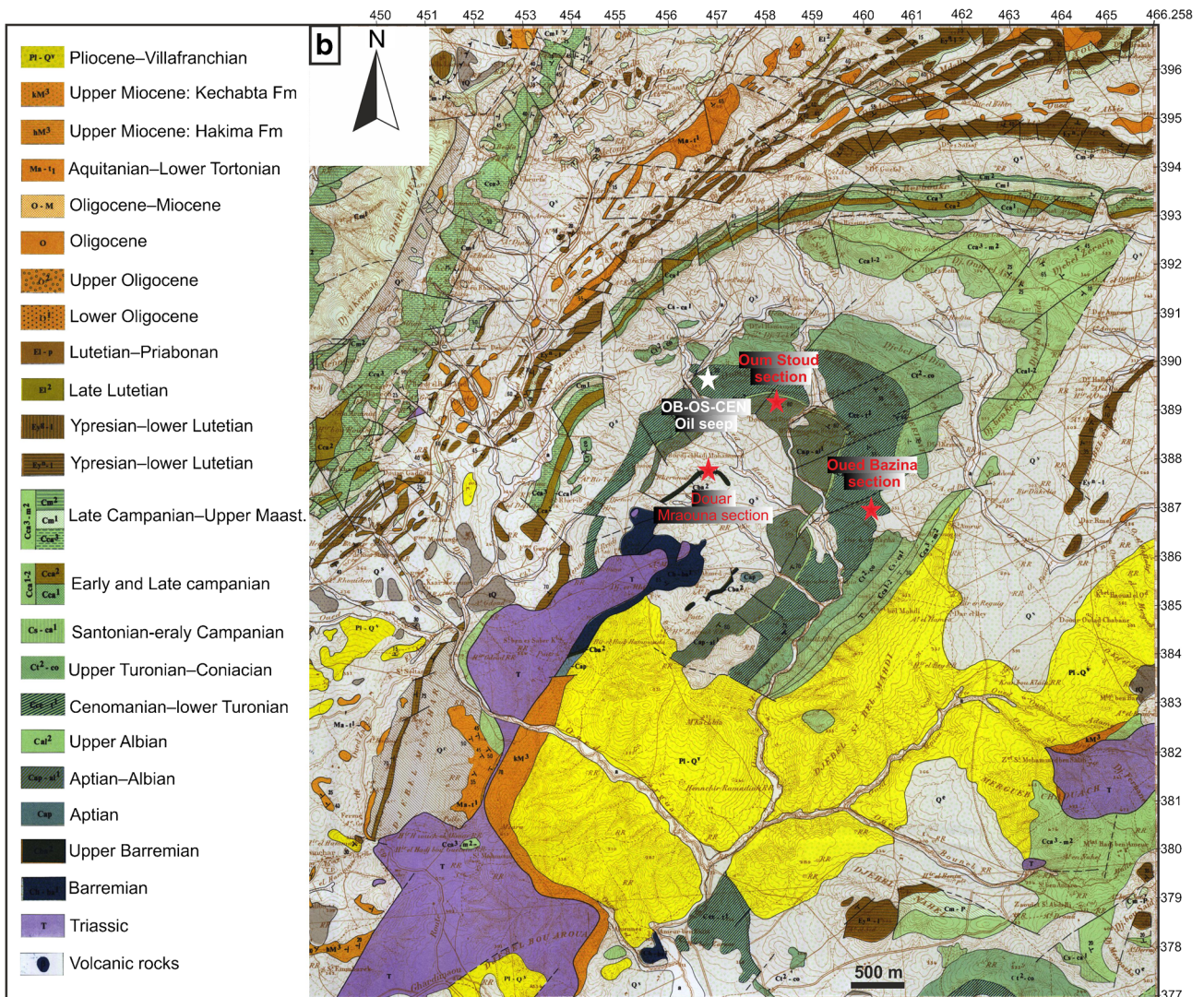
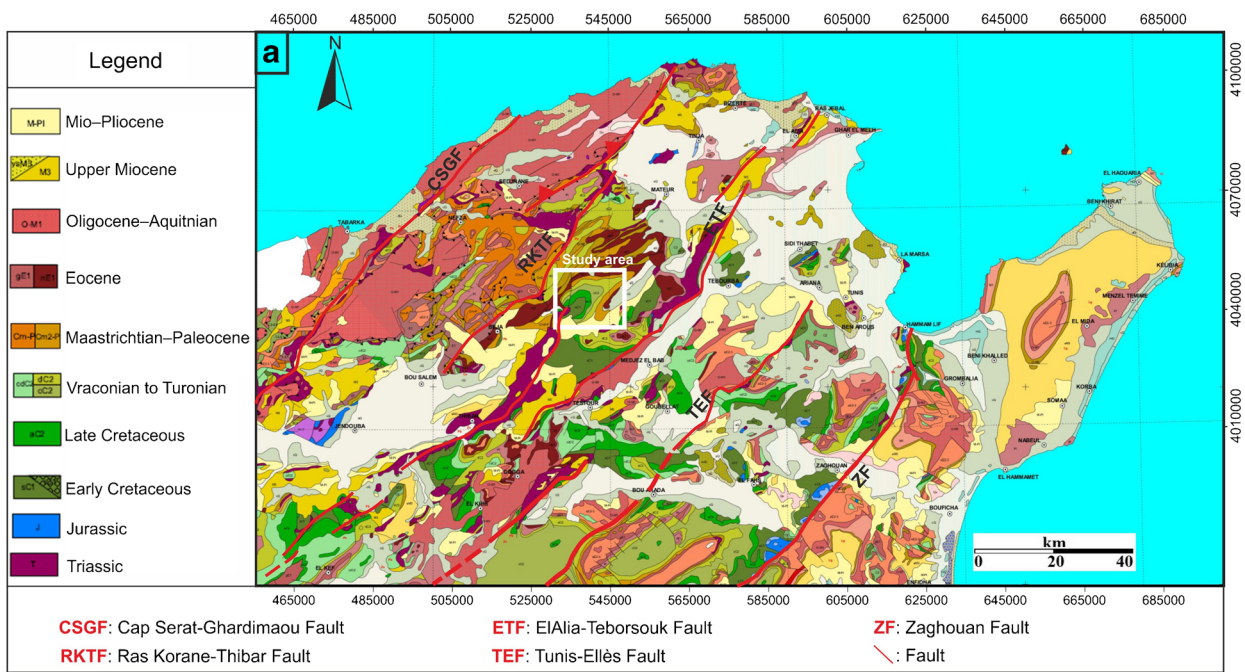
In the area of Douar Mraoua, the early Cretaceous succession (upper Barremian) is well exposed and represented by 11.5 m thick succession made up of black laminated limestones and marls. The section base is dominated by dark grey marls overlain by laminated black marly–limestones and laminated limestones plates. The uppermost part of the section is represented by massive black limestones rich in ammonites (Fig. 3). Stratigraphically, this section appears to be time-equivalent of the upper Barremian aged succession studied by Talbi et al. (2019) and Soua (2016) which thought to be deposited in the collapsed areas of the basin due to tilting by normal E–W faults.

### Albian organic-rich strata: Fahdene Formation of Oum Stoud section

In the Oum Stoud section, the Albian succession is mainly dominated by thick series of black marls and shales with subordinate dark grey carbonate beds. The sedimentation begins with dark grey–black limestones and argillaceous limestones, overlapped by levels of nodular black limestones of massive aspect (8–15 m thick) with thin marls parting intervals which in turn capped by dark grey marls with intercalation of massive black limestones and marly limestones. The middle part of the section is 130 m thick and includes mainly grey marls. The upper part (upper Albian) includes black marly intervals interbedded with limestones and massive marly limestones that locally contain glauconite (Fig. 3). This section displays difference with the Albian succession in the Tajerouine section which is considered to have more basinal characteristics (Chihaoui et al. 2010).

### Cenomanian–Turonian organic-rich strata: Bahloul Formation

The Cenomanian–Turonian organic-rich strata of the Bahloul Formation are well exposed in the Oued Bazina area and subdivided into three lithological units ( $U_1$ ,  $U_2$  and  $U_3$ ). The lower unit ( $U_1$ ) is dominated by black laminated limestone



◀ **Fig. 2** a Geological maps of northern Tunisia showing the major faults of northern Tunisia, the position of the studied sections and the recorded oil seep sample outcropping in the NE of Thibar salt dome structure (modified from Melki et al. 2012). b Detailed geological map of the studied area boxed in Fig. 2a (Biely et al. 1982)

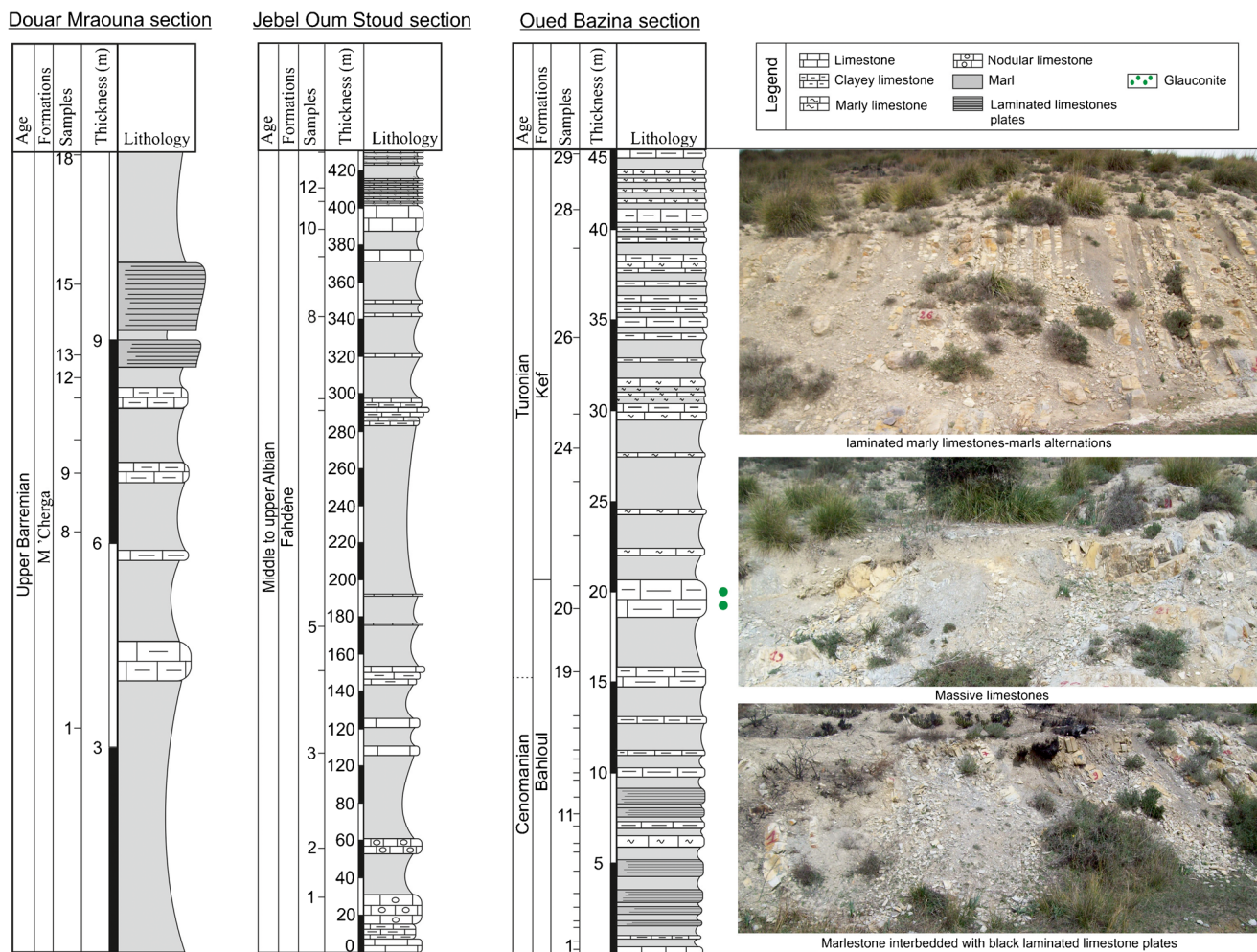
plates with thin marls and laminated marly limestone partings. The medium unit (U<sub>2</sub>) is composed of black marls enclosing subordinate black limestone beds. The uppermost unit (U<sub>3</sub>) tends to be dominated by laminated and massive limestones.

## Materials and methods

### Rock-Eval pyrolysis

Total rock geochemistry analysis was conducted on 52 samples (Table 1) that were collected from three outcrop sections represented by Douar Mraouna section (upper Barremian succession: ten samples), Oum Stoud section (Albian succession: 13 samples) and Oued Bazina section (Cenomanian–Turonian

succession: 29 samples). They were collected from different lithologies including marlstones, limestone beds and black laminated limestones plates. Samples from Oued Bazina (OBZ) and Douar Mraouna (DM) sections were collected and analysed using Rock-Eval-6 method (Espitalié et al. 1985; Espitalié et al. 1977; Tissot and Welte 1984; Lafargue et al. 1998; Behar et al. 2001), while samples from Oum Stoud (OS) section were provided by Tunisian Oil Company (ETAP) from internal report (Belayouni 1994). The adopted sampling is 1–3 m intervals spacing with a high-resolution sampling in the black shale interval 0.3–0.7 m thick. Several parameters were determined from Rock-Eval-6 pyrolysis method such as total organic carbon (TOC) contents, S<sub>1</sub> (free hydrocarbons fraction), S<sub>2</sub> (fraction of hydrocarbons released by thermal cracking) and maximum pyrolysis temperature (T<sub>max</sub>, corresponding to maximum kerogen cracking measured at the top of the S<sub>2</sub> peak which indicates the maturity level or stage of source rocks). Other parameters have been determined by Rock-Eval-6 such as hydrogen index (HI) and oxygen index (OI). Units and formula of the mentioned parameters above are represented in Table 2. These parameters



**Fig. 3** Lithological succession of upper Barremian, middle to upper Albian and Cenomanian–Turonian organic-rich strata

**Table 1** Rock-Eval-6 (Espitalié et al. 1985) analysis of upper Barremian, Albian and Cenomanian–Turonian samples from Douar Mraouna, Jebel Oum Stoud and Oued Bazina, respectively

Section	S <sub>1</sub> (mg HC/g R)	S <sub>2</sub> (mg HC/g R)	PP (mg HC/g R)	TOC (wt%)	T <sub>max</sub> (°C)	HI (mg HC/g TOC)	OI (mg CO <sub>2</sub> /g TOC)
Upper Barremian succession: Douar Mraouna section							
DM18	0.15	3.28	3.43	1.22	447	269	16
DM15	1.98	10.47	12.45	3.8	448	276	8
DM13	0.24	5.36	5.6	3.28	442	163	38
DM12	1.17	9.76	10.93	3.88	445	252	19
DM11	0.32	6.08	6.4	3.65	441	167	41
DM10	0.1	2.52	2.62	1.72	444	147	42
DM9	1.14	12.02	13.16	4.92	445	244	18
DM8	0.35	4.94	5.29	2.63	445	188	34
DM7	0.7	7.97	8.67	3.24	443	246	15
DM1	0.05	0.83	0.88	0.58	444	143	55
Albian succession: Oum Stoud section							
OS13	0.33	3.71	4.04	0.78	434	424	–
OS12	0.12	5.02	5.14	0.95	436	528	–
OS11	0.14	5.22	5.36	1.15	436	454	–
OS10	0.03	1.3	1.33	0.64	434	203	–
OS9	0.01	0.4	0.41	0.6	440	67	–
OS8	0.11	7	7.11	2.03	436	345	–
OS7	0.02	0.97	0.99	0.49	442	198	–
OS6	0.02	2.5	2.52	0.8	438	313	–
OS5	0.01	0.3	0.31	0.35	436	86	–
OS4	0.03	0.77	0.8	0.25	438	308	–
OS3	0.02	0.52	0.54	0.22	432	236	–
OS2	0.02	0.92	0.94	0.41	440	224	–
OS1	0.43	0.4	0.83	0.24	436	167	–
Cenomanian–Turonian succession: Oued Bazina							
OBZ29	0.08	5.06	5.14	1.23	433	411	51
OBZ28	0.04	3.68	3.72	0.92	431	400	36
OBZ27	0.11	6.79	6.9	1.25	429	543	28
OBZ26	0.01	0.27	0.28	0.11	443	245	145
OBZ25	0.02	1.51	1.53	0.7	434	216	69
OBZ24	0.02	0.87	0.89	0.35	434	249	57
OBZ23	0.11	5.62	5.73	1.74	432	323	50
OBZ22	0.76	20.04	20.8	3.03	428	661	12
OBZ21	0.31	11.76	12.07	1.93	429	609	24
OBZ20	0.48	15.28	15.76	2.23	429	685	23
OBZ19	0.34	11.43	11.77	1.78	430	642	30
OBZ18	0.9	22.05	22.95	2.98	429	740	21
OBZ17	0.38	14.47	14.85	2.03	430	713	27
OBZ16	1.6	44.06	45.66	6.3	427	699	16
OBZ15	0.24	12.07	12.31	2.39	431	505	41
OBZ14	1.85	38.76	40.61	5.24	427	740	17
OBZ13	0.47	17.92	18.39	2.51	432	714	18
OBZ12	1.26	32.24	33.5	4.16	430	775	10
OBZ11	2.38	51.46	53.84	6.76	430	761	9
OBZ10	1.56	40.74	42.3	4.24	429	961	4
OBZ9	1.26	32.38	33.64	4.01	430	807	8
OBZ8	1.13	26.37	27.5	3.49	432	756	8
OBZ7	0.2	8.05	8.25	1.76	432	457	45
OBZ6	0.01	1.2	1.21	0.44	441	273	107
OBZ5	0.05	5.25	5.3	1.38	436	380	72
OBZ4	0.6	20.3	20.9	3.78	422	537	27
OBZ3	0.01	0.08	0.09	0.12	434	67	367
OBZ2	0.01	0.13	0.14	0.14	437	93	143
OBZ1	0.1	4.76	4.86	1.38	437	345	72

are given throughout an applied cycle where pyrolysis started at 300 °C allowing getting S<sub>1</sub> peak, and then the sample was heated to 650 °C obtaining the so-called S<sub>2</sub> peak. The cycle is completed by combustion of the residual rock recovered after pyrolysis, and the sample was heated up to 850 °C, and the S<sub>3</sub>

peak (CO<sub>2</sub> released) is determined. The oxidation (combustion) started isothermally at 400 °C (Espitalié et al. 1985; Behar et al. 2001). TOC is determined by the CO and CO<sub>2</sub> during pyrolysis and oxidation stages, and it is expressed in weight percentage (wt%).

**Table 2** Various parameters obtained by Rock-Eval pyrolysis

Parameters	Unit	Formula	Name
$T_{max}$	°C	$TpS_2 - \Delta T_{max}$	$T_{max}$
RC CO	wt%	$[S_4CO \times 12/28]10$	Residual org. carbon (CO)
TR		$S_1 \text{ HC Exp.}/S_1 + S_2$	Transformation ratio
PC	wt%	$[(S_1 + S_2) \times 0.83] + [S_3 \times 12/44] + [(S_3CO + S_3'CO/2) \times 12/28]$	Pyrolysable org. carbon
PI		$S_1/(S_1 + S_2)$	Production index
RC CO <sub>2</sub>	wt%	$[S_4CO_2 \times (12/44)]/10$	Residual org. carbon (CO <sub>2</sub> )
RC	wt%	$RC \times CO + RC \times CO_2$	Residual org. carbon
TOC	wt%	$PC + RC$	Total organic carbon
HI	mg HC/g TOC	$(S_2 \times 100)/TOC$	Hydrogen index
OI	mg CO <sub>2</sub> /g TOC	$(S_3 \times 100)/TOC$	Oxygen index

### Gas chromatography coupled to mass spectrometry method

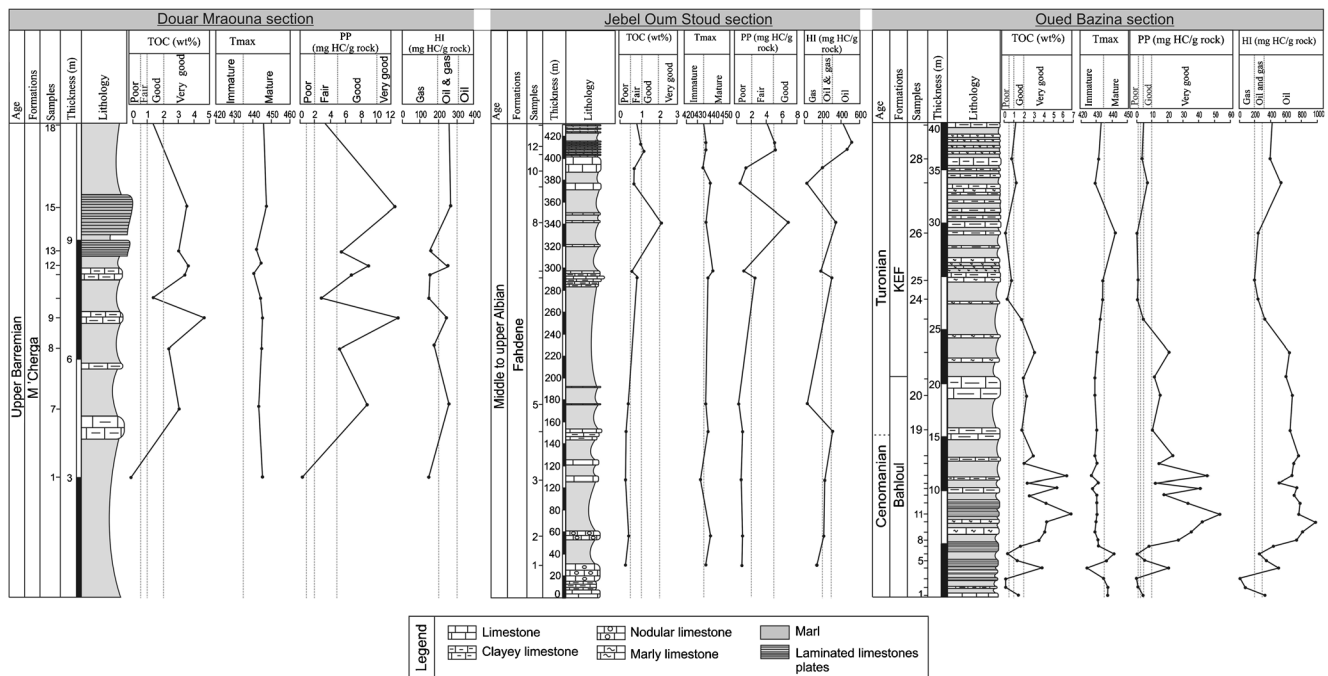
Seven samples were analysed through gas chromatography coupled to mass spectrometry (GC/MS) which were firstly well prepared in order to be passed to GC/MS analysis (6 samples from the main Cretaceous source rocks and one oil seep sample recorded in the fractured Cenomanian strata). The bitumen fraction was extracted with organic solvent (dichloromethane: CH<sub>2</sub>Cl<sub>2</sub>), and then liquid chromatography with a single column was performed to recuperate saturate, aromatic and polar fractions. Saturate biomarkers were determined using Agilent 7890A gas chromatograph interfaced to an Agilent 5975 quadrupole mass spectrometer. The GC was equipped with split/splitless injection system, operated in split mode, and DB-IMS fused

silica capillary column of 30 m length, 0.25 mm inner diameter and 0.25 µm film thickness. Helium was used as a carrier gas with a flow rate of 1 ml/min. The oven temperature used to analyse saturates was programmed from 50 °C (hold 2 min) to 170 °C (5 min) to 300 °C at 1.5 °C/min. The mass spectrometer was operated in the electron impact mode at electron energy of 70 eV and a source temperature of 230 °C.

### Results

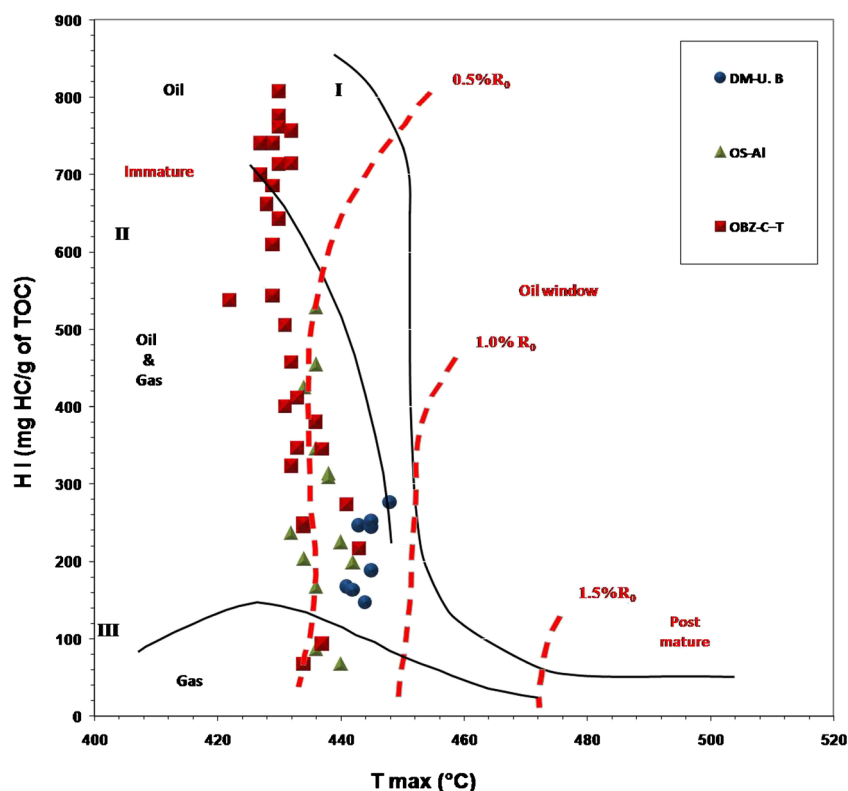
#### Rock-Eval pyrolysis analysis of the upper Barremian

The organic content of the upper Barremian samples in Douar Mraoua section presents high organic contents (TOC)



**Fig. 4** TOC, total organic carbon, wt%;  $T_{max}$ , temperature at maximum of S2 peak; HI, hydrogen index= $S_2 \times 100/TOC$ , mg HC/g TOC; PP, petroleum potential yield = $S_1+S_2$  (mg/g)

**Fig. 5** HI vs.  $T_{max}$  diagram of the main Cretaceous source rocks. (DM-U. B: upper Barremian section; OS-AI: Albian section; OBZ-C-T: Cenomanian–Turonian section)



ranging from 0.58 to 4.92% (Table 1). The pyrolysis derived  $S_1$  and  $S_2$  values range from 0.1 to 0.44 mg/g and 1.06 to 9.30 mg/g, respectively. The majority of values indicate fair to good source rock potential. Few samples have mostly poor potentiality than other samples. Most samples present high petroleum potential values reaching 13.16 mg HC/g rock.  $T_{max}$  values of upper Barremian range from 441 to 448 °C with a mean value of 441 °C suggesting that the samples are early to mid-mature for oil generation. The hydrogen index ( $HI = S_2 \times 100/TOC$ , mg HC/g TOC) corresponds to the peak  $S_2$  normalized for the TOC content and serves as indication of kerogen types (Tissot and Welte 1984; Peters 1986). The hydrogen index (HI) values range from 147 to 276 mg/g, reflecting that this formation is dominated by gas-prone kerogen with 62.5% of the representative samples occur under 200 mg HC/g TOC indicating a type III organic matter, and 37.5% of samples are between 200 and 300 mg HC/g TOC indicating a mixed organic matter type II/III (Fig. 5).

### Middle to upper Albian organic-rich strata of Jebel Oum Stoud section

The organic richness (TOC) of the middle–upper Albian (Fahdene Formation) varies from poor to fair (Fig. 4b) with values ranging from 0.24 to 1.15% (Table 1). Only one sample (OS8) is rated good to very good. The pyrolysis derived “ $S_1$ ”

values range from 0.01 to 0.43 mg/g, and the remaining potential “ $S_2$ ” values range from 0.30 to 5.22 mg/g (Table 1; Fig. 4b), indicating that they are of poor to fair source rock potential (Fig. 4b).  $T_{max}$  range from 434 to 442 (Table 1; Fig. 4b), indicating an immature source rocks. HI values range from 67 to 454 mg/g (Table 1; Fig. 4b), indicating that the Fahdene Formation ranges from oil-prone to gas-prone kerogen with 41% of the representative samples occurring between 300 and 700 mg HC/g TOC and indicating a marine planktonic organic matter (type II), and 34% of the studied samples are derived from continental organic matter (type III), while 25% of the samples present type II/III organic matter (Fig. 5)

### Cenomanian–Turonian source rock

Samples collected from Cenomanian–Turonian facies (Bahoul Formation) outcropping in Oued Bazina area present high TOC values reaching 6.76% with a mean value of 2.82% (Table 1; Fig. 4c).  $T_{max}$  values of the Cenomanian–Turonian organic-rich strata are very low and did not exceed 430 °C in this area. HI vs.  $T_{max}$  diagram (Fig. 5) shows that most of the samples collected from these facies present high HI values exceeding 300 mg HC/g which indicate that this source rock is very rich in oil-prone kerogen and containing essentially marine planktonic organic matter (type II).



**Table 3** Geochemical data from Cretaceous organic-rich strata analysed using liquid chromatography and GC/MS.

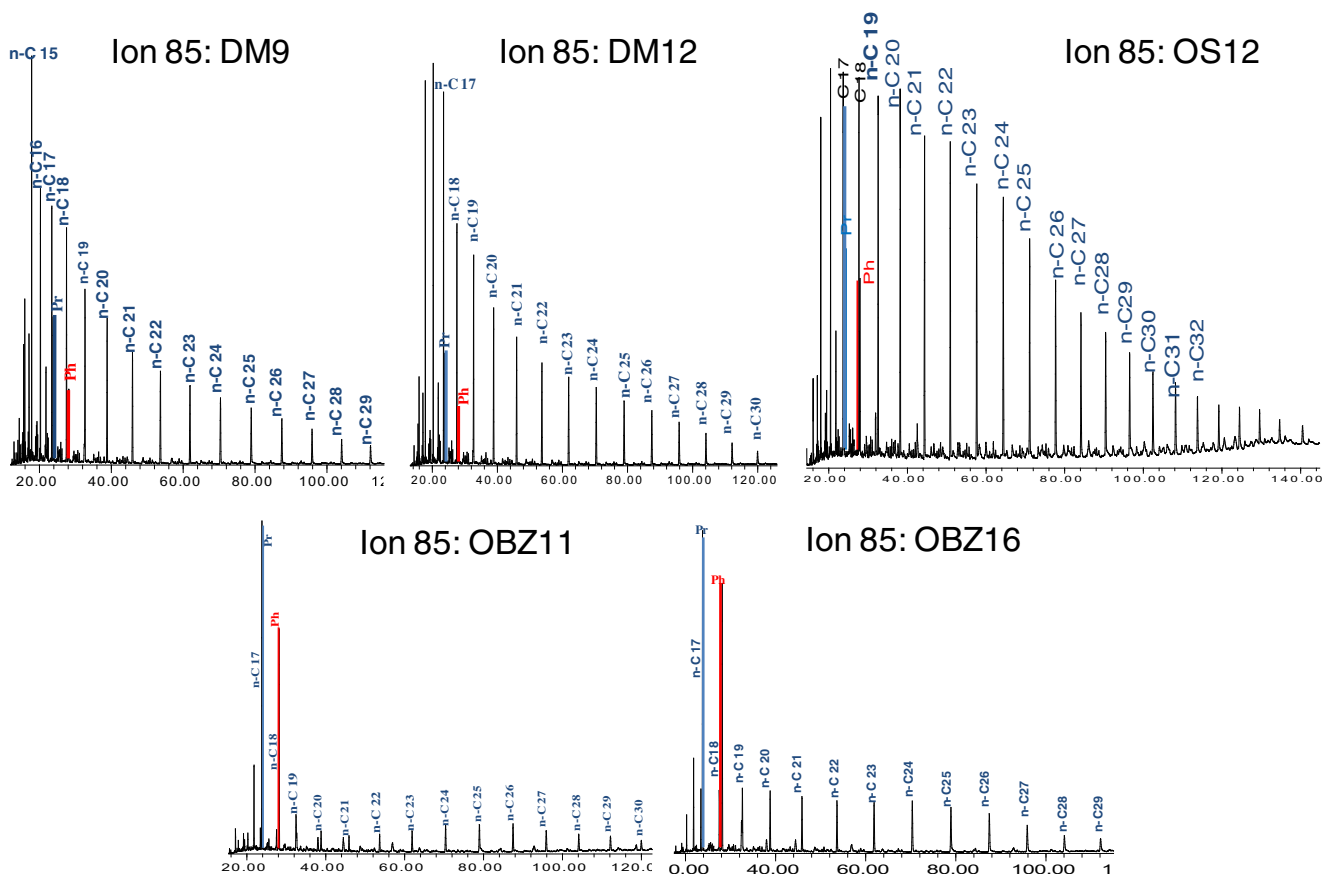
Parameters	DM12	DM9	OS12	OBZ11	OBZ16
Saturates (%)	45	34	47	27	28
Aromatics (%)	25	44	44	42	38
NSO compounds	30	22	9	31	34
Pr/Ph	2.5	2.48	2.21	1.8	1.4
C <sub>19</sub> /(C <sub>19</sub> + C <sub>23</sub> )	0.38	0.38	0.34	0.04	0.05
C <sub>24</sub> Tet/(C <sub>24</sub> Tet + C <sub>23</sub> TT)	0.31	0.32	0.22	0.26	0.29
C27 (%)	33	34	31	27	27
C28 (%)	31	30	29	38	39
C29 (%)	36	36	39	34	34

### Molecular composition of the Cretaceous organic-rich strata

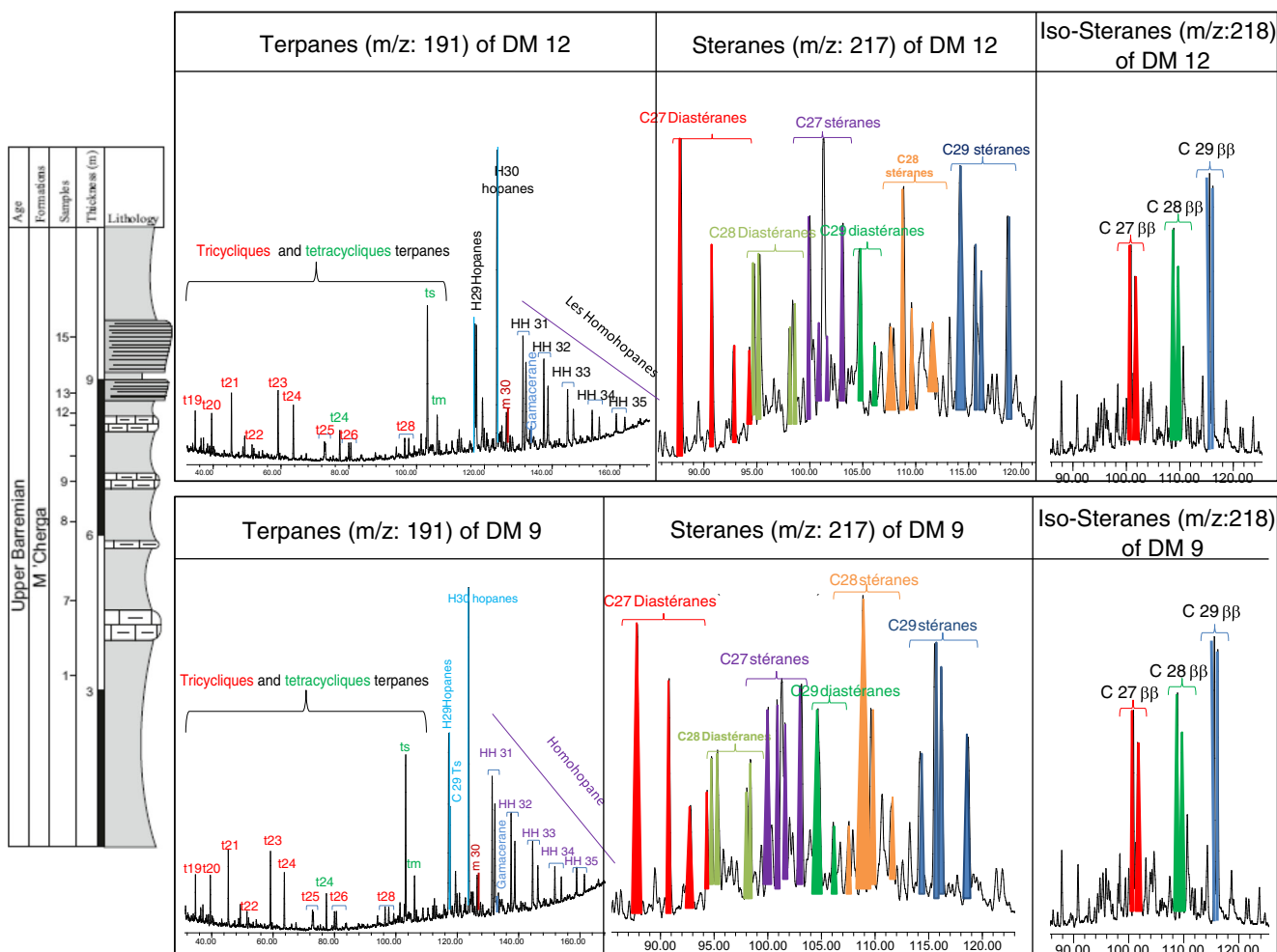
Samples collected from the upper Barremian, Albian and Cenomanian–Turonian organic-rich facies outcropping in the NE of Thibar diapir (northern Tunisia) are rich in organic extracts or bitumen. The organic extracts are dominated by saturate and aromatic hydrocarbons over the NSO compounds

(resins and asphaltenes). Values of total hydrocarbons represent 77% of the organic extracts for upper Barremian facies, 91% for the Albian facies and 68% for Cenomanian–Turonian facies (Table 3).

Molecular biomarkers lead to the reconstruction of the geological history of the organic-rich rocks. In fact, they consist of molecular fingerprints that survive after all diagenetic phenomena. According to their molecular structures, there are several types of biomarkers. The saturate fraction of upper Barremian and Albian samples (DM9, DM12 and OS12) shows unimodal n-alkane distribution ranging from n-C<sub>15</sub> to n-C<sub>30+</sub> and maximizing around n-C<sub>17</sub> and n-C<sub>18</sub>. Moreover, n-alkane distribution of Cenomanian–Turonian samples (OBZ11 and OBZ16) exhibits an equal concentration (Fig. 6). In all studied samples of the Cretaceous series, the Pristane (Pr) dominates the Phytane (Ph) as indicated by the relatively high Pr/Ph ratio ranging from 1.4 to 2.5. The upper Barremian and Albian organic-rich facies are marked by a dominance of n-C<sub>18</sub> and n-C<sub>17</sub> over Pr and Ph, respectively, with lowest value recorded in the upper Barremian facies (Ph/n-C<sub>18</sub> ratio = 0.42). However, in the Cenomanian–Turonian samples (OBZ11 and OBZ16), the Pr and Ph dominate their respective n-alkanes.



**Fig. 6** Mass chromatograms showing the distribution of n-alkane and iso-alkanes ( $m/z$  85) of DM9 and DM12 (upper Barremian), OS 12 (Albian) and OBZ11 and OBZ16 (Cenomanian–Turonian) samples



**Fig. 7** Mass chromatograms showing the distribution of terpanes ( $m/z$  191), steranes ( $m/z$  217) and iso-steranes ( $m/z$  218) relative to the upper Barremian source rock outcropping in the study area

The steranes ( $m/z$  217 and 218) and terpanes ( $m/z$  191) were used to determine the geochemical characteristics of each source rock such as the origin of organic matter, thermal maturity, lithology, depositional environment and biodegradation. The abundance of regular steranes over diasteranes ( $m/z$  217; Figs. 7, 8, and 9) suggests a carbonate lithology approved by the important concentrations of tetracyclic terpane T24 (Palacas et al. 1984; Connan and Dessort 1987). The diasterane/sterane ratio is generally influenced by both mineral matrix and thermal degradation (Tissot and Welte 1984). Samples collected from the upper Barremian and Cenomanian–Turonian facies (DM12, DM 9, OBZ11 and OBZ16) show, on the ion 217 mass spectrum, a relative predominance of steranes compared with diasteranes.

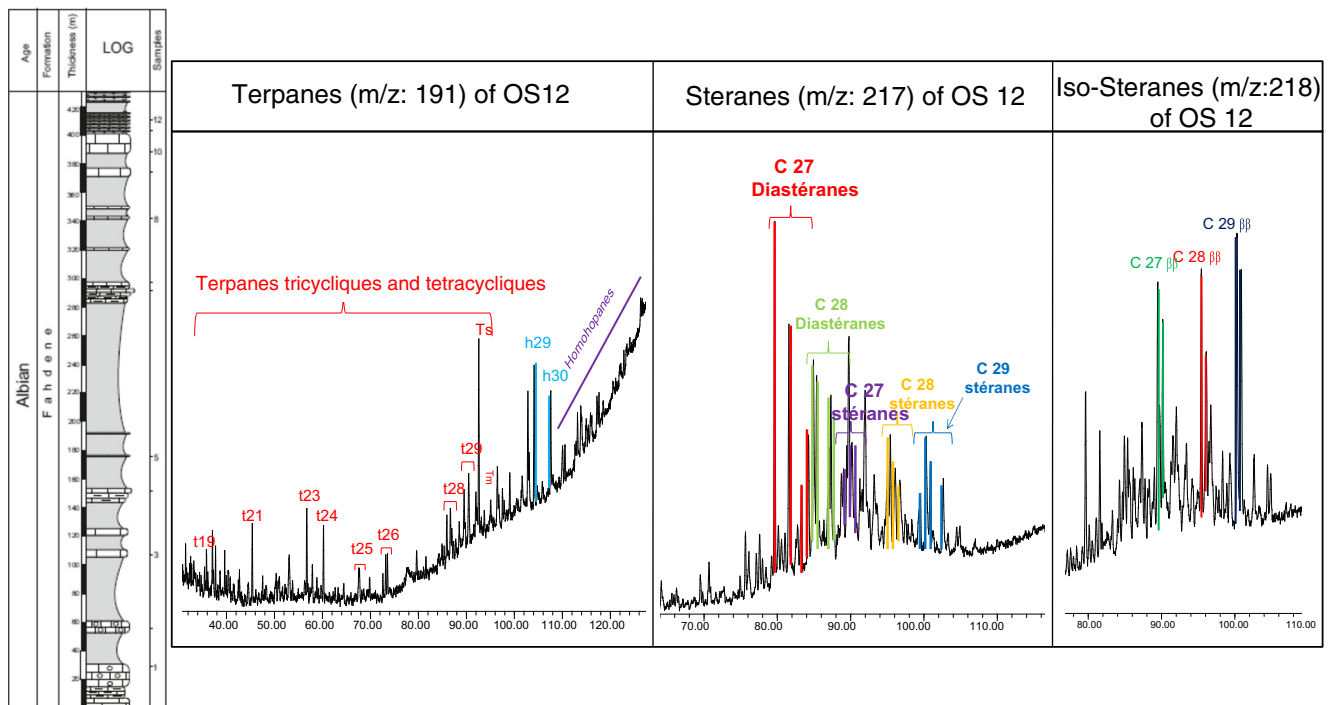
The  $C_{27}$ -,  $C_{28}$ - and  $C_{29}$ -steranes can be used as indicators of the depositional environment and the organic matter origin (Moldowan and Mc Caffrey 1995). The sterane distributions of the upper Barremian and Albian samples (DM12, DM9 and OS12) show a predominance of  $C_{29}$ -steranes over  $C_{28}$ - and  $C_{27}$ -steranes. However, OBZ11 and OBZ16 samples

collected from the Cenomanian–Turonian black shales exhibit a predominance of  $C_{28}$ -steranes over  $C_{27}$  and  $C_{29}$  homologous.

$C_{27}$  18 $\alpha$ -22,29,30-trisnorneohopane (Ts) is thermodynamically more stable than  $C_{27}$  17 $\alpha$ -22,29,30-trisnorhopane (Tm) (Peters and Moldowan 1993), and the ratio increases with increasing maturity. Samples from upper Barremian and Albian facies (DM12, DM9 and OS12) exhibit the predominance of Ts over Tm with Ts/Tm ratio values ranging from 3.42 to 3.81 for upper Barremian samples and of 2.91 for Albian sample. In contrast, samples from the Cenomanian–Turonian facies (OBZ11 and OBZ16) show a predominance of Tm over Ts with very low Ts/Tm ratio of 0.17 and 0.19, respectively.

### Oil seep molecular analysis

The bimodal n-alkane distributions of the OB-OS-CEN oil seep sample indicate its sourcing from a rock that presents a marine organic matter origin with some continental



**Fig. 8** Mass chromatograms showing the distribution of terpanes ( $m/z$  191), steranes ( $m/z$  217) and iso-steranes ( $m/z$ : 218) relative to the Albian source rock outcropping in the study area

contribution. The analysed sample displays a moderate predominance of Pristane compared with Phytane with a Pr/Ph ratio of 1.9 indicating a sub-oxic depositional environment (Fig. 10a). This is supported by the homohopane ( $C_{31}$ – $C_{35}$ HH) distribution of the ion 191 mass spectra. Additionally, there is a relative abundance of diasteranes compared with steranes indicating that the source rock of the studied oil sample is relatively rich in clay fraction. (Fig. 10b). Gammacerane comes from the reduction of tetrahymina (Venkatesan 1989). The low concentration of gammacerane in OB-OS-CEN oil seep sample indicates deep marine environment characterized by normal salinity (Niu et al. 2018) with gammacerane/hopane ratio value of 0.05.

## Discussion

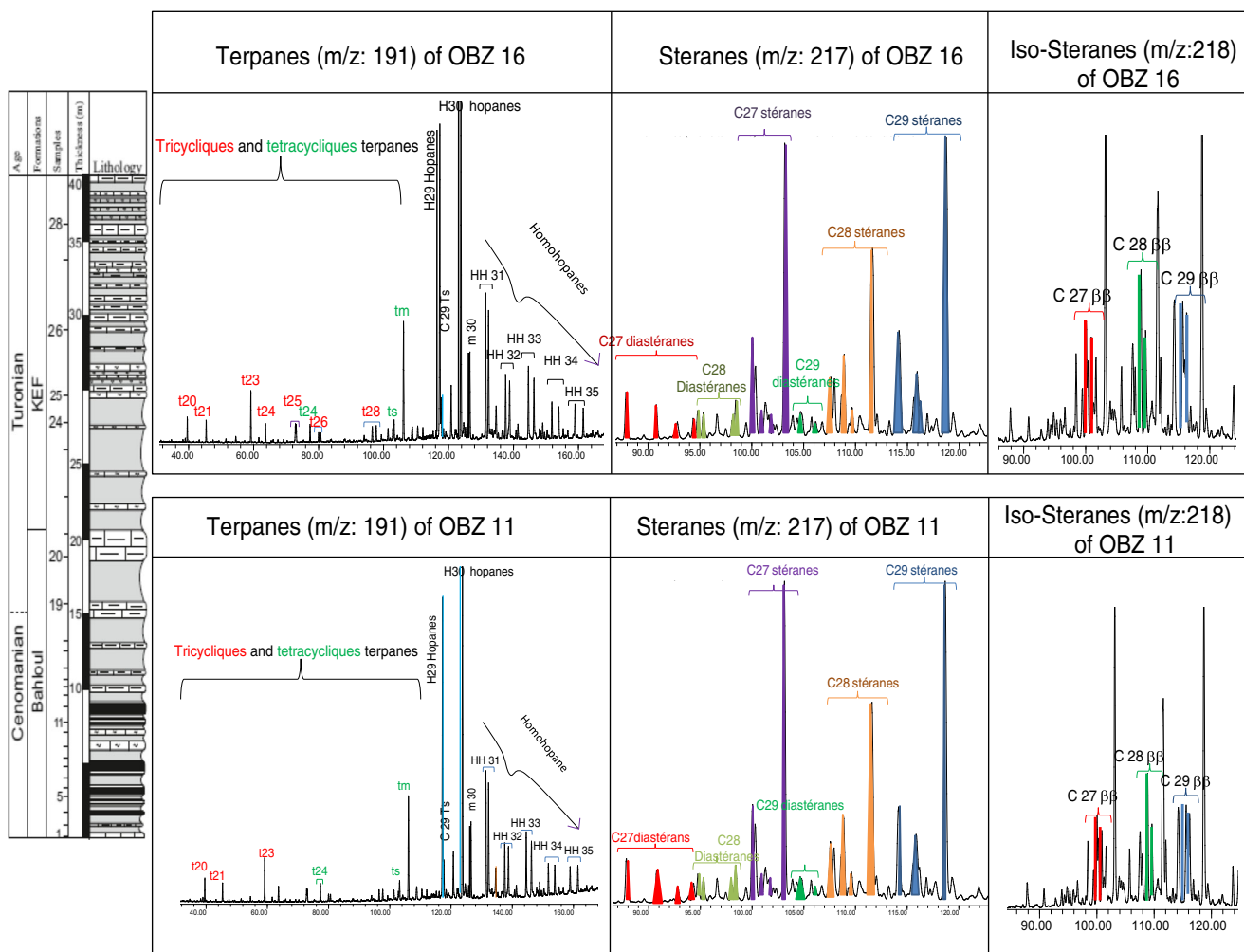
### Organic richness and generative petroleum potential

Rock-Eval pyrolysis analysis of the Cretaceous organic-rich strata outcropping in Oued Bazina area, NE of Thibar diapir, was used to evaluate the organic matter richness and the petroleum potential (PP).

Based on the petroleum potential vs. TOC diagram (Fig. 11), samples from the three studied Cretaceous source rocks split into two groups: (1) a highly organic-rich group with an excellent petroleum potential

represented by the Cenomanian–Turonian strata (Bahloul Formation) and the upper Barremian black laminated limestone plates and marls and (2) a moderate organic-rich group with poor to fair petroleum potential represented by the Albian succession (Fahdene Formation) of Oum Stoud section.

Our results concur with available published geochemical studies (Layeb 1990; Belayouni 1994; Abbassi 2008; Chaari 2002; Affouri et al. 2013; Ben Fadhel et al. 2014; Ayari 2015; Talbi et al. 2019) undertaken on the upper Barremian, Albian and Cenomanian–Turonian organic-rich strata in the salt dome zone and in the Medjerda valley (Figs. 12 and 13). Nevertheless, the Cenomanian–Turonian anoxic event in the salt dome area displays a wide variation of TOC values (TOC values are varying from 1.69 to 2.85% with a mean value of 2.4% and a PP ranging between 4 and 20.28 kg of HC/ton of rock) (Fig. 13) that is directly related to the particular geometry of the basin during the lower to middle Cretaceous epochs. Within this frame, it is thought that the structural framework was controlled by NE–SW faults (Boltenhagen 1985; Chihi 1995) that generated half-graben structures characterized by thick sedimentary prisms on the collapsed side of normal faults and reduced ones on their resistant side (Talbi et al. 2019). These half-grabens have acted as restricted mini-basins favouring the water stagnation, the accumulation and preservation of the organic matter and consequently the development of locally euxinic conditions in the bottom waters.



**Fig. 9** Mass chromatograms showing the distribution of terpanes ( $m/z$  191), steranes ( $m/z$  217) and iso-steranes ( $m/z$  218) relative to the Cenomanian–Turonian source rock outcropping in the study area

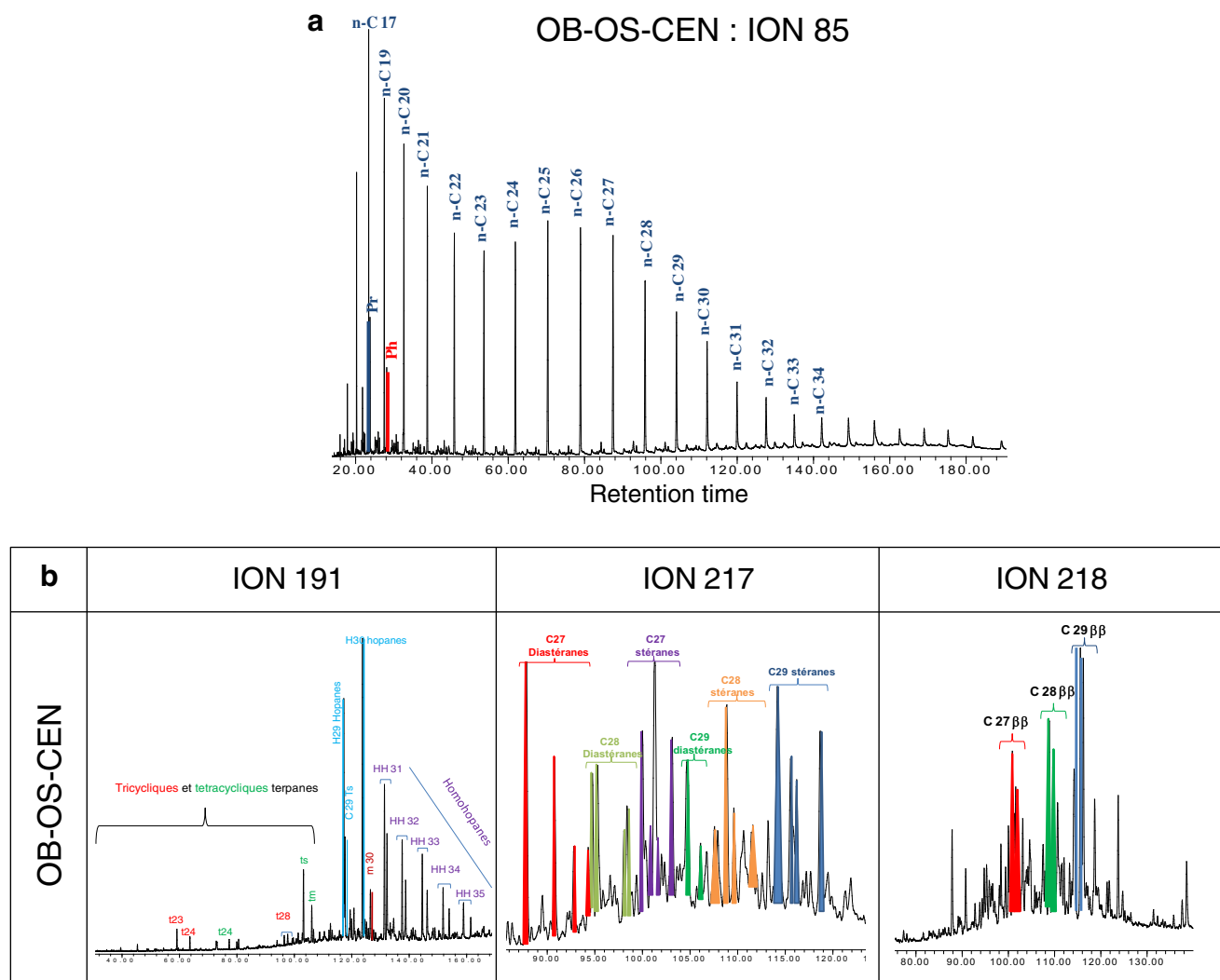
The record of the anoxic event is evidenced by the high amount of organic matter included in the black laminated limestone plates, black limestones and marls in the Bahloul Formation in Oued Bazina.

In several areas located above or close to the Triassic salt domes, the early and middle Albian deposits are commonly missing, and only the late Albian and Vraconian series are present (paleohigh related hiatus). This is the case of Oum Stoud section where the early Albian is lacking. The organic richness (TOC) of the middle–upper Albian (Fahdene Formation) varies from poor to fair (Fig. 4b) with values ranging from 0.24 to 1.15% (Table 1). Only one sample (OS8) is rated good to very good. The comparison of our results with available data (Belayouni 1994; Abbassi et al. 2010; Chaari 2002; Ben Fadhel et al. 2014; Talbi et al. 2019) show that in the salt dome zone, the TOC values are ranging from 0.52 to 1.1% with a mean of 0.8%, while the petroleum potential is varying between 1.08 and 2.82 kg of HC/ton rock which is considered as poor to fair PP.

### Lithology, depositional environment and kerogen type

The predominance of steranes comparatively with diasteranes in the upper Barremian (DM9 and DM12) and Cenomanian–Turonian (OBZ11 and OBZ16) samples indicates that the organic-rich facies are composed of carbonates containing some argillaceous fraction (Figs. 7 and 9). In contrast, the analysed Albian sample (OS12) shows the predominance of diasteranes compared with steranes and therefore suggesting a more clastic source rock (Fig. 8). Upper Barremian samples (DM9, DM12) contain more clastic fraction than Cenomanian–Turonian samples (OBZ11 and OBZ16) (Fig. 14).

The low concentration of gammacerane in samples collected from upper Barremian, Albian and Cenomanian–Turonian (DM9, DM12, OS12, OBZ11 and OBZ16) indicates that the studied Cretaceous source rocks were deposited in a deep marine environment with normal salinity. The upper



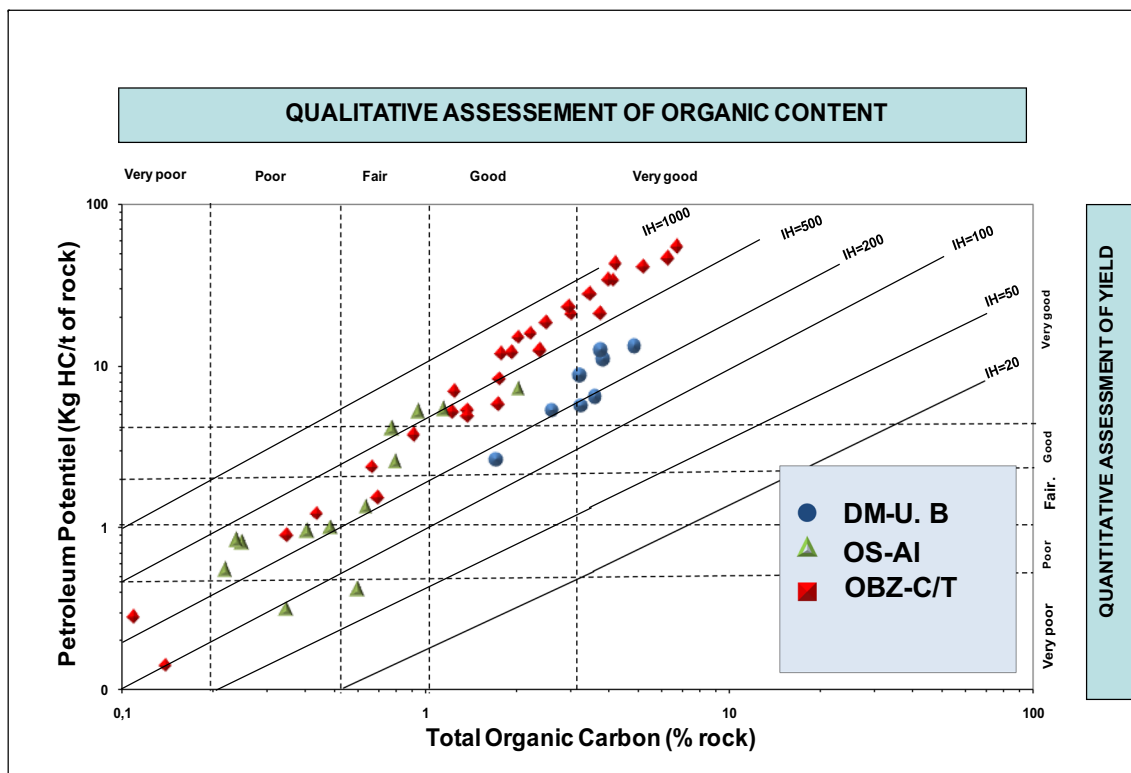
**Fig. 10** Mass chromatograms showing the distribution of n-alkanes and iso-alkanes ( $m/z$  85), terpanes ( $m/z$  191), steranes ( $m/z$  217) and iso-steranes ( $m/z$  218) of OB-OS-CEN oil seep sample

Barremian samples show a regular decreasing of homohopanes from C<sub>31</sub> to C<sub>35</sub> which is characteristic of a sub-oxic depositional environment. However, the irregular decrease of C<sub>31</sub>–C<sub>35</sub> hopanes of samples from Cenomanian–Turonian source rock (OBZ11 and OBZ 16) indicates a sub-oxic to anoxic depositional environment (Figs. 7, 8, and 9). The Pristane/Phytane ratio tends to be high in more oxidizing environments and low in strongly reducing ones (Powell and McKirdy 1973). Pr/Ph ratio indicates a sub-oxic depositional environment for the upper Barremian and Albian successions and a sub-oxic to anoxic environment for the Cenomanian–Turonian organic-rich strata with values of 2.48–2.5, 2.21 and 1.4–1.8, respectively (Table 3).

Tricyclic terpanes have also been associated with the alga *Tasmanites* (Revill et al. 1994). Abundant tricyclic terpanes are attributed to a common feature of a freshwater environment (Kruege et al. 1990). The diagram of C19/(C19 + C23)Tt

vs. C24TT/(C24Tt + C23Tt) shows that samples from the upper Barremian, Albian and Cenomanian–Turonian facies are deposited in marine environment (Fig. 15). A comparison with other localities suggests that results of upper Barremian are similar to those of Jebel Ammar and Jebel Ressas (Elkhazri et al. 2009). Cenomanian–Turonian facies preserved a type II organic matter signature in the salt dome zone (Affouri et al. 2013).

The distribution of the C27, C28 and C29 ( $\alpha\alpha$ 20R) regular steranes can serve as a facies parameter (Shanmugam 1985; Killops and Killops 2005). Indeed, C27 sterols are derived from algae, while C29 sterols are synthesized from land plants (Volkman 1986). Samples from the Cretaceous source rocks have shown a marine algal input (Fig. 16). The absence of terrestrial inputs in the upper Barremian, Albian and Cenomanian–Turonian source rocks concurs with results of Hallek et al. 2020.



**Fig. 11** Qualitative and quantitative evaluations of the main mid-Cretaceous source rocks. (MT-U. B: upper Barremian section; OS-AI: Albian section; OBZ-C-T: Cenomanian–Turonian section)

## Thermal maturity

Assessment of thermal maturity of the three source rocks outcropping in the NE of Thibar salt dome is determined by Rock-Eval pyrolysis,  $T_{\max}$  values and  $T_s/T_m$  distribution. The upper Barremian and Albian facies (DM9, DM12 and OS12 samples) exhibit the predominance of  $T_s$  over  $T_m$  and therefore indicating a mature stage. The upper Barremian facies  $T_s/T_m$  ratio (3.81) indicates more advanced thermal maturity level compared with the Albian facies ( $T_s/T_m$  ratio = 2.91). However, the Cenomanian–Turonian facies (OBZ11 and OBZ16 samples) show a very low  $T_s/T_m$  ratio indicating an immature stage (Fig. 9). The  $C_{29} \alpha\alpha S/C_{29} \alpha\alpha R$  vs.  $C_{29} \beta\beta R/C_{29} \alpha\alpha R$  diagram shows that samples from upper Barremian source rock (DM12 and DM9) present high maturity level compared with samples from Albian (OS12) and Cenomanian–Turonian source rocks (OBZ11 and OBZ16) (Fig. 17).

The  $T_{\max}$  values recorded in the Cenomanian–Turonian samples organic-rich strata are very low (429–437 °C) confirming that this source rock is immature.  $T_{\max}$  values of the Albian succession in Oum Stoud section range from 434 to 442 °C indicating that the Albian source rock is early mature

to mature.  $T_{\max}$  values recorded in Douar Mraouna section (441–448 °C) indicate that the upper Barremian source rock is mature with high respect to the oil window (Table 1; Fig. 4a).

The integration of our results on thermal maturity with published data indicates clearly that the thermal maturity degree is fairly low (immature) toward the eastern part (Oued Bazina, Oued Faouar and Ech Cheid) as indicated by the relatively low  $T_{\max}$  values which range from 431 to 439 °C, while toward the west (Sfa Boubaker, Jebel Sraissif and Jebel Hdidia), the Fahdene (Albian) and the Bahloul (Cenomanian–Turonian) source rocks are mature to late mature as indicated by the relatively high  $T_{\max}$  values varying between 440 and 446 °C according to the results of Rock-Eval pyrolysis from the outcrop sections studied by several authors (Chaari 2002; Ben Fadhel et al. 2014; Affouri et al. 2013). The increase of the thermal maturity appears to be related to the high rate of sedimentation and subsidence driven by regional tectonic movements along NW–SE, NE–SW and east–west trending normal faults (Soua et al. 2009). However, in the NE part, the Cretaceous outcrops of Oued Bazina deposited close to the paleohighs as the result of the Triassic salt dome movement since the lower Cretaceous have not buried

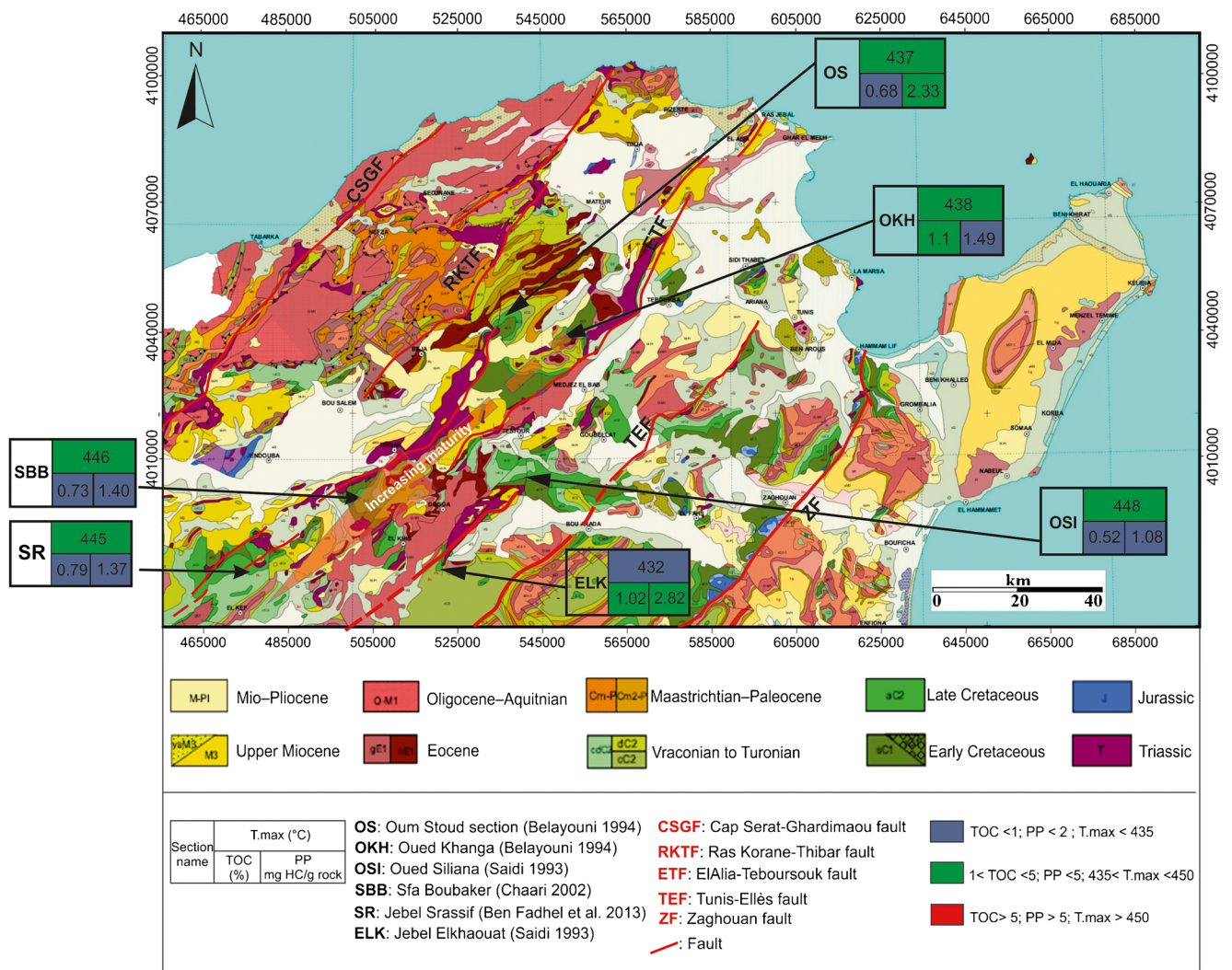


Fig. 12  $T_{max}$ , TOC and petroleum potential distribution of Albian source rock in northern Tunisia

enough to produce oil. Nevertheless, and despite their position close to Ech Cheid salt dome, the Albian and Cenomanian–Turonian organic-rich facies of the Oued Siliana section (Figs. 12 and 13) show high degree of thermal maturity (448 °C and 442 °C). Comparison of the geochemical results of the Cenomanian–Turonian and Albian facies according to a NE-SE transect extending from Oued Faouar, Oued Siliana to Oued Bazina (Figs. 12 and 13) shows that the thermal maturity increases near the Triassic salt structure, whereas in the inter-diapiric domains, we notice a decrease in maturity (e.g. Oued Faouar and Oued Bazina sections). This arrangement prompted us to look for the link between high thermal maturity and salt dome structures. In this context, Downs (2012) showed that salt domes strongly increase the parameter of the temperature in the sedimentary deposits, which has an impact on the maturity of the organic

matter and on the time of hydrocarbon generation. Thus, the cases of Oued Siliana can be included in this framework where the Bahloul Formation (Oued Siliana) is located close to the Jebel Echid diapir that increased the heat flow because of the thermal anomaly surrounding Jebel Ech Cheid salt structure.

### Oil/source rock correlation

Oil/source rock correlation using biomarker analysis permit to determine the source of the single oil seep recorded in the Cenomanian faulted limestone beds. Three samples (DM12-U.B, OS12-AI and OFO-C/T) collected from Cretaceous source rocks (upper Barremian, Albian and Cenomanian–Turonian) have been used for this correlation. Regarding the low maturity stage of Cenomanian–Turonian samples of Oued Bazina area, we provided

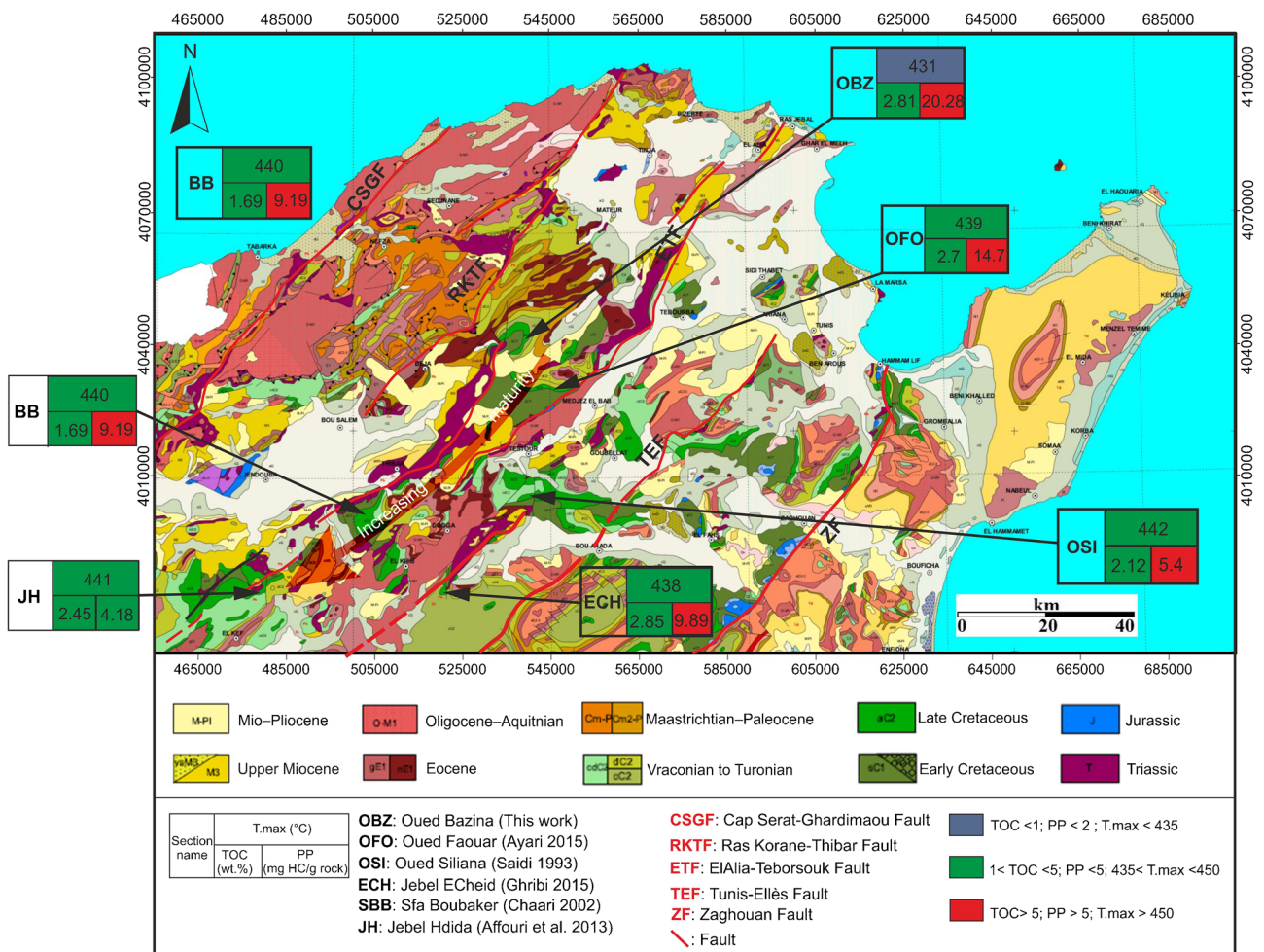
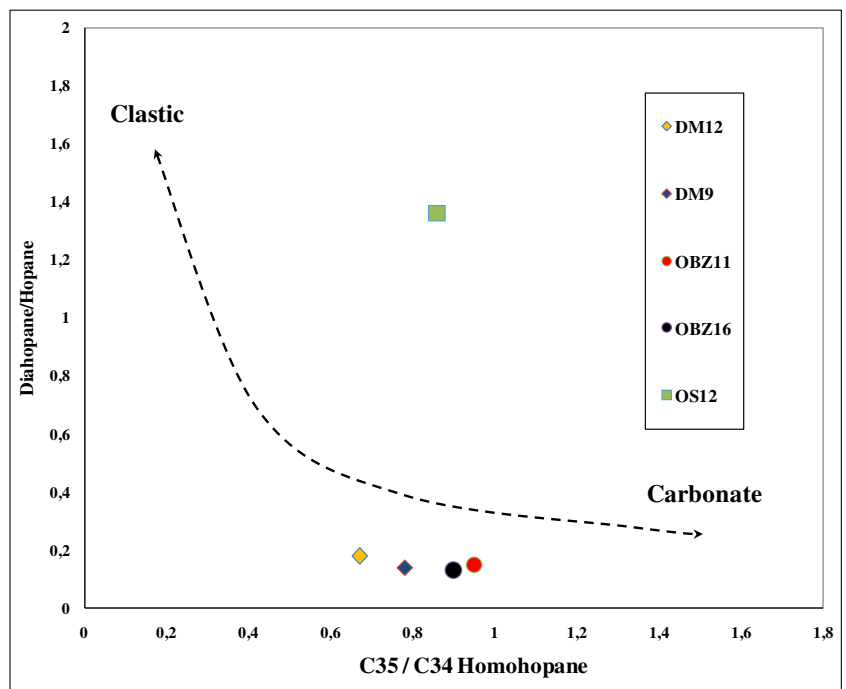


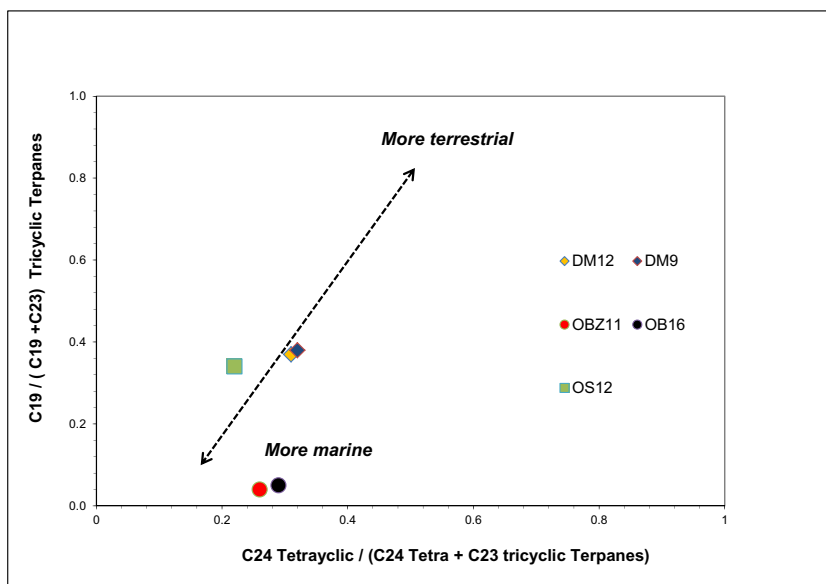
Fig. 13  $T_{max}$ , TOC and petroleum potential distribution of Cenomanian–Turonian source rock in northern Tunisia

Fig. 14 Diahopane/hopane vs.  $C_{35}/C_{34}$  homohopane diagram of samples from upper Barremian section (DM9, DM12), Albian section (OS12) and Cenomanian–Turonian section (OBZ11, OBZ16)





**Fig. 15** C19/(C19 + C23) vs. C24TT/(C24TT + C23TT) diagram

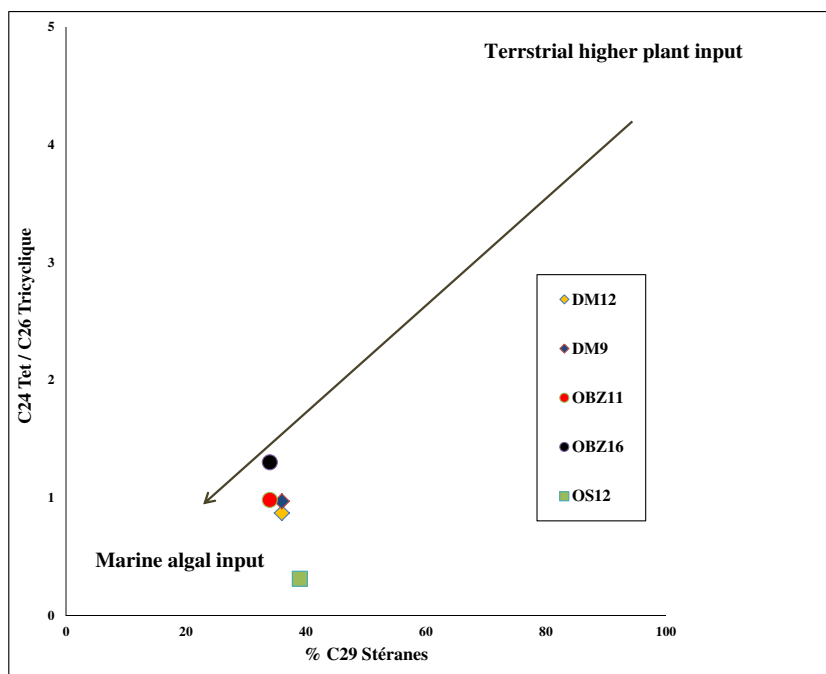


another sample from Oued Faouar region (OFO-C-T). Sterane, terpane and hopane distributions exhibit similar values for upper Barremian source rock (DM12-U. B) and OB-OS-CEN oil seep. This result indicates that the single oil seep recorded in Oued Bazina area “OB-OS-CEN” was sourced from the upper Barremian source rock (Figs. 18 and 19).

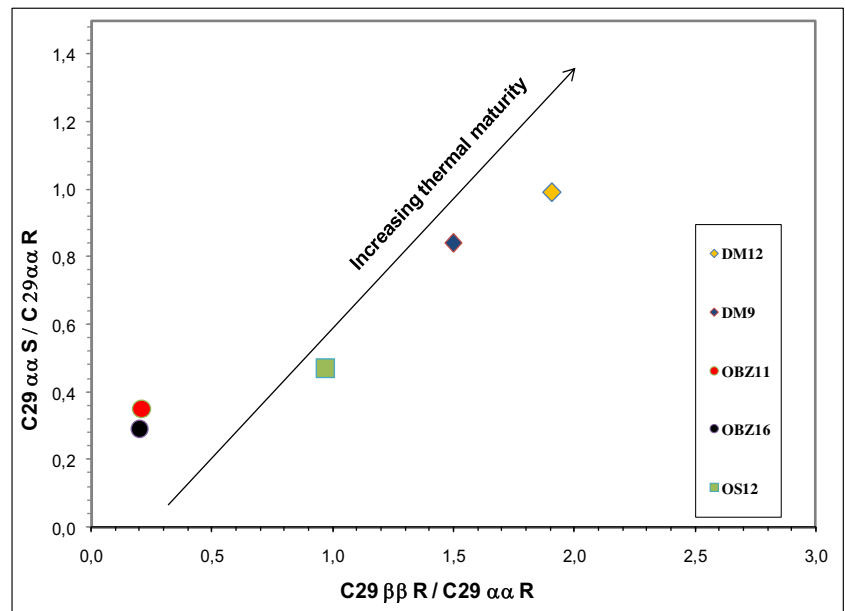
### Conclusion

Results from organic geochemistry study of the Cretaceous organic-rich deposits outcropping in northern Tunisia using Rock-Eval pyrolysis and biomarkers showed good potential source rocks especially for upper Barremian and Cenomanian–Turonian where their TOC and petroleum

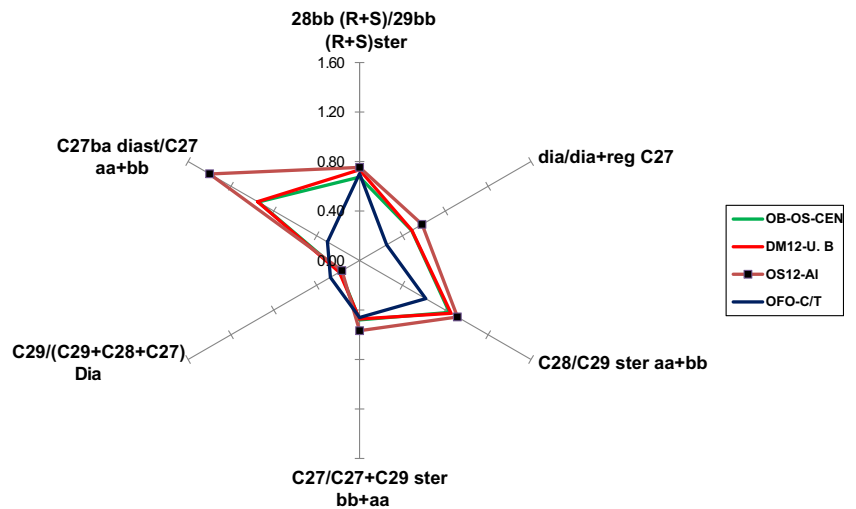
**Fig. 16** C24TT/C26T vs. %C29 regular sterane diagram of samples from upper Barremian section (DM9, DM12), Albian section (OS12) and Cenomanian–Turonian section (OBZ11, OBZ16)



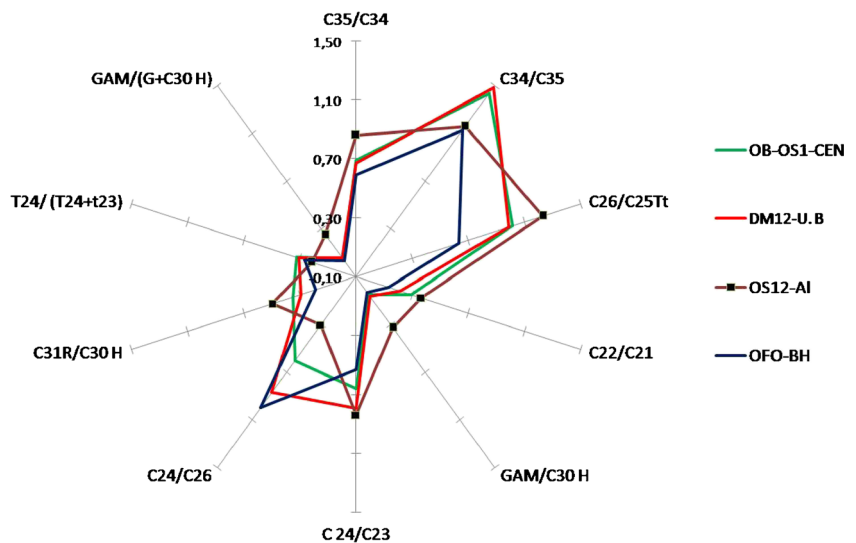
**Fig. 17** The C29  $\alpha\alpha$ S/C29  $\alpha\alpha$ R vs. C29  $\beta\beta$ R/C29  $\alpha\alpha$ R diagram of samples from upper Barremian section (DM9, DM12), Albian section (OS12) and Cenomanian–Turonian section (OBZ11, OBZ16)



**Fig. 18** Oil/source rock correlation based on sterane and diasterane values. (OB-OS-CEN: Cenomanian oil seep sample; DM12-U. B: upper Barremian sample; OS12-Al: Albian sample; OFO-C/T: Cenomanian–Turonian sample)



**Fig. 19** Oil/source rock correlation based on terpane, tricyclique and hopanes values (OB-OS-CEN: Cenomanian oil seep sample; DM12-U. B: upper Barremian sample; OS12-Al: Albian sample; OFO-C/T: Cenomanian–Turonian sample)



potential values are very high. Organic matter of the main Cretaceous source rocks is mainly deposited in marine environment controlled by algal input. Only the upper Barremian source rock presents relatively high maturity level (oil window) and must have generated an appreciable quantity of hydrocarbons (mainly oil), and some of them could have been detected in the study oil seep. Despite the large sediments overlaying the Turonian facies,  $T_{\max}$  values revealed low maturity level for Albian and Cenomanian–Turonian source rocks in Oued Bazina area.

The correlation of oil/source rocks requires us to reconsider with a particular interest the upper Barremian black shales for oil and gas exploration.

Our present study provides a new understanding of this new important petroleum source rock (PP reaching 13 kg HC/T Rock, over 7–11 m of thickness) around Thibar diapir as well as a new insight into an active petroleum system in northern Tunisia.

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