First evidence of the early cretaceous oceanic anoxic events (MBE and OAE1a) in the southern Tethyan margin (NE-Tunisia): biostratigraphy and shale resource system

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Abstract

The Jebel Oust region (north-eastern Tunisia) recorded two levels of marine black shale in the Lower Cretaceous marly series. Geodynamic evolution, biostratigraphic and Rock–Eval analyses allow classifying those black shales as unconventional shale oil resource systems that were deposited during two oceanic anoxic events: the Middel Barremian Event "MBE" and the Early Aptian Event "OAE1a". Paleogeographic evolution highlights two transgressive–regressive cycles: the first one is Valanginian–Early Barremian, and the second is Late Barremian–Early Aptian. Each black shale deposit occurs at the end of the transgression that coincides with the highest sea level. During the Barremian–Aptian interval, sedimentation was controlled by extensional faults in a system of tilted fault blocks which were reactivated several times. Kerogen is of type I, II origin in black shales and of type III origin in marls. Tmax values indicate "oil window" stage. Average transformation ratio is around 67% and 82%, respectively, in the Lower Aptian and Middel Barremian source rock related to the relatively high thermal maturity degree due to the deep burial of the later. Estimated initial hydrocarbon generation potential is moderate to high. Oil saturation index records an "oil crossover" indicating expelled and migrated hydrocarbons from the organic-rich to the organic-poor facies. The petroleum system of the two mature source rocks with a high hydrocarbon generation potential enclose all elements characterizing a "shale oil hybrid system with a combination of juxtaposed organic-rich and organic-lean facies associated with open fractures".

Keywords Oceanic anoxic events · Shale oil system · Open fracture · Rock–Eval pyrolysis

Introduction

The Middel Barremian Event (MBE) black shales were detected in the north Tethyan margin and took place during a short episode (Coccioni et al. 1992, 2003, 2006; Sprovieri et al. 2006; Godet et al. 2006; Bodin et al. 2009; Malkoc and Mutterlose 2010; Yilmaz et al. 2012). However, the Early Aptian anoxic event ("Selli Event": "OAE1a") took place for a long time (5 Ma) and was generally interpreted as a

high-productive event well correlable, taking into account both global and local factors (Bralower et al. 1994; Leckie et al. 2002; Luciani et al. 2006; Graziano 2013; Huck et al. 2013; Moosavizadeh et al. 2014). This study presents the first recording of the oceanic anoxic event of Mid-Barremian outcrops in the southern Tethyan margin. While OAE1a, which is already well known, extended to the northern Tunisia (Talbi 1991; Elkhazri et al. 2013; Godet et al. 2014; Soua 2016; Talbi et al. 2018). A geochemical and biostratigraphic approach is used to invest organic compounds distribution through the source rocks as well as characterizing and dating the two oceanic anoxic events MBE and OAE1a recorded in the Barremian–Aptian series which outcrop in the locality of Jebel Oust (NE Tunisia) (Fig. 1). An estimate of the primary organic content, transformation ratio, retained and expelled hydrocarbons in the diverse sedimentary facies will make possible characterization of the various elements that constitute the shale oil resource system such are migration, reservoir rocks and traps. Finally, geodynamical and

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Fig. 1 a Geographic regional situation; b Northeast Tunisian geologic map, c detailed geologic map of Jebel Oust region and d simplified geologic map of study area showing OAH and AA' section location

palaeogeographical sketch are put forward to identify the factors controlling both the deposit and the evolution of the unconventional resource system.

Geological setting

Jebel Oust region is located about thirty kilometers south of Tunis City (Fig. 1). It's a part of the Tunisian eastern Atlas, and it is represented by predominantly clayey outcrop sediment of the "Tunisian furrow". The NE-SW structure was acquired during recent compression phases (Ben Ayed 1986). The core of the anticline is represented by Jurassic neritic limestones (Sidi Khralf Formation) (Memmi 1989) that were deposited in an inner platform environment. During the Jurassic, the Jebel Oust region belonged to the stable external platform of eastern Tunisia, included in the pelagian domain (Salaj 1978). Its disconnection and total collapse at the upper Jurassic were the consequence of the sinister strike-slip faulting on Europe relative to Africa (Ben Ayed 1986). The post-Triassic sedimentary analysis shows that the maximum depth of the sedimentary basin occurred at the Valanginian-Barremian interval (M'Cherga formation). Clay-marl series deposited during this interval are about three times thicker than their equivalent of the Voconian basin. So, the region constituted, at the early Cretaceous, the umbilicus of the eastern branch of the Tunisian furrow with about 3000 m thick of M'Cherga formation (Fig. 2) (Jauzein 1957; Ferjani et al. 2018).

The basin was affected at different times by rifting resulting in E-W and N-S strike-slip faults associated with tilted fault blocks. At the Lower Cretaceous, the Jebel Oust region is being pulled apart subsiding megastructure (Guiraud and Maurin 1991). It was an example of extensional down faulted depression in the Tunisian furrow during the Jurassic-Terminal Barremian interval. The Barremo-Aptian period was characterized by a distensive NE-SW tectonic episode (Castany 1954; Chihi et al. 1984; Saïd et al. 2011; Gharbi et al. 2015) which affects the depth and the inner architecture (Fig. 3).

During Aptian-Albian interval (Fahdene formation), the Tunisian furrow was marked by distensive tectonic activity reactivated old deep faults and created halokinetic activities that contributed to Triassic salt dome genesis at the core of the current anticline structures (Smati 1986; Perthuisot et al. 1988) (Fig. 3). This produced inequalities, anisopaque sedimentation, unconformities, dismantling breaches and reef constructions (Burollet 1991; Perthuisot et al. 1988). The geological cross-section of the study area: OAH (Oust Ain Hamam) is located in the Jebel Oust area. Its GPS coordinates are N 360 30 '59.3; E 0100 05 '38.9. This geologic section crosses a clay limestone series of Barremian–Aptian age, with about 900-m-thickness (Fig. 1d).

Fig. 2 General log showing main geologic formations outcropping in Jebel Oust region (modified, from Memmi 1989)

Fig. 3 Schematic NW–SE cross-section showing black shales related to MBE and OAE1a within tilted blocks, guiding the Jebel Oust Barremian–Aptian source rock sedimentation

Paleogeographic setting

The depositional environment during the Barremian–Aptian interval has been controlled by the structure of the seafloor that has affected types, distribution and thickness of sedimentary facies (Jauzein 1957; Fournie et Pacaud 1973; M'rabet 1981; Memmi 1989) (Fig. 4).

Two sedimentary mega-sequences were deposited during the Lower Cretaceous transgressive–regressive cycles (Memmi 1989). The first mega-sequence was regressive during Tithonic–Early Barremian interval. It filled the collapsed areas by predominate clay-fine quartz deposits that has been carried by southern turbidity currents. Those arrived from central Tunisia which was occupied by a prograding deltaic complex. The second mega-sequence which is transgressive–regressive with bathyal deep sea sedimentation started at Middle Barremian and ended in Gargasian (Fig. 2). This sequence is formed by argillite with fine

sandstone pasts and clay-carbonates (Jauzein 1957; Buroillet et al. 1983; Memmi 1989). In the southern domain of Tunisian furrow, the Kairouan Island remained emerged during the entire Early Cretaceous interval, and the North–South Axis was less subsiding and recognized condensed levels (Jauzein 1967; Marie et al. 1982, Buroillet et al. 1983; Memmi 1989) (Fig. 4).

Materials and methods

After geological survey of the Early Cretaceous outcropping strata in Jebel Oust region, a geological cross-section (OAH) was constructed through the Barremian–Aptian series (Figs. 2 and 5). A systematic sampling is carried out in order to study both the released microfauna and the organic geochemistry analysis. The sampling becomes very tight near the Barremian–Aptian limit (Fig. 5). To

Fig. 4 Palaeogeographic map of Tunisia in the lower cretaceous (Marie et al. 1982)

study the released microfauna, we used the standard technique of washing the marls. Planktic, benthic foraminifers and Ostracods are observed with a Scanning Electron Microscope. The biostratigraphic approach is based on the contribution of these species which are of great interest in dating the Barremian–Aptian series. The petroleum shale resource system evaluation of the Barrem–Aptian series was realized based on organic geochemistry analyses that were conducted on 15 rock samples using Rock–Eval 6 programmed pyrolysis. This technical method (Espitalié et al. 1977; Sykes and Snowdon 2002) measures the total organic carbon (TOC), and the compounds released during organic matter pyrolysis of 100 mg of finely ground sediment under an inert atmosphere (Helium) and by programming the heating temperature from 180–850 °C at a rate

of 25 °C/mn. A pyrogram showing three peaks related to three organic compounds is provided: the S1 and S2 peaks correspond, respectively, to the free hydrocarbon quantities released at 180 °C and to the potential hydrocarbons obtained by thermal cracking of the insoluble organic matter (kerogen) at a temperature ranging between 180 and 550 °C. The S3 peak represents the oxygen compounds in organic matter, and the maximum temperature (Tmax) measured at the summit of the S2 peak corresponds to the kerogen thermal maturity degree. Finally, the highest temperature (up to 550 °C) led to determine TOC content. Several parameters are calculated using S1, S2 and S3 such as hydrogen index ($HI = (S2 \times 100)/TOC$ in mg HC/g TOC), oxygen index ($OI = (S2 \times 100)/TOC$ in mg CO₂/g TOC) and S2/S3 ratio. All these parameters are classical and are

Fig. 5 Detailed geologic section showing vertical distributions of microfauna specimens in Jebel Oust Barremian–Aptian series (OAH section)

used for petroleum source rock characterization. Parameters used as indicators for production and oil expulsion have been invented, and others have changed indication such as retained hydrocarbons ($RHC = S1 + S2$), hydrocarbon generation potential ($HGP = S2$), Oil saturated index

($OSI = (100 \times S1)/TOC$). Original organic parameters are estimated (TOC_0 and $S2_0$) according to Jarvie et al. (2007) method ($TR = (HI_0 - HI)/HI_0$).

Fig. 6 Variations of TOC, Hydrocarbon generation potential (HGP), Oil saturation index (OSI) and Tmax showing "oil crossover" in Jebel Oust Barremian–Aptian series

Results

Biostratigraphy study of the Barremian–Aptian interval

Biostratigraphy of the geological cross-section (Fig. 4) refers to microfaunal assemblage of planktic and benthic foraminifers and age determinations are cross-checked with the documented Ammonite zonation for the Barremian–Aptian stages carried out by Memmi (1989). The bottom of the section begins with gray marls that are surmounted by alternations of consolidated marls, limestones and clayey limestones with gray marls intercalation which contain the following planktic foraminifera: *Gorbachikella kugleri* and *Praehedbergella sigali* and benthic foraminifera: *Lenticulina ouachensis ouachensis*, *L. ouachensis bartensteini*, *L. nodosa*, *L. eichenbergi*, *Gavelinella barremiana*, *Dentalina oligostegia*, *Praedorothia hechti*, *Epistomina sp* with rare ostracods, radiolarian and echinoderms. This series belongs to the *Holcodiscus caillaudi* ammonite zone that marks the Early Barremian.

On this series, are deposited clayey limestones including six black shale levels. The last two levels are silicified, ending by an eroded surface which could affect the high submarine forms (horsts). This may be induced by tectonic instability occurring during a short time at the end of the Early Barremian. Following this eroded surface, a hard nodular pure limestone bar was settled with about 100 m thickness that serve as a regional landmark formation. Those limestones show slumps on curved slip surfaces and did not provide characteristic fauna. They were attributed to the Early Late Barremian (Memmi 1989).

A thick greenish marly series (200 m) with thin quartz beds ends the Barremian. These series contain the following faunal association: *Gorbachikella depressa*, *Gorbachikella kugleri*, *Praehedbergella sigali*, *Hedbergella sp*, *Globigerina hotervica*, *Lenticulina ouachensis ouachensis*, *L. ouachensis bartensteini*, *L. nodosa*, *L. eichenbergi*, *Gavelinella barrigia*, *Dentalina olivia*, *D. D. gracilis* with some ostracods: *Cytherella sp*, *Cythereis sp* and *C. arabica*. This microfauna association and the ammonite *Lepidoceras puzosianum* mark the Late Barremian (Salaj 1980;

Memmi 1989; Bellier and Moullade 2002; Chida-Ben Haj Ali 2005).

The early Aptian is represented at its bottom by gray-black marls interspersed by clay limestones which support brown marls, surmounted by two levels of black shale and dark clayey limestones. This series provides the following planktic fauna association: *Gorbachikella kugleri*, *Praehedbergella sigali*, *Praehedbergella aptiana* with a benthic fauna composed of *Lenticulina ouachensis*, *L. ouachensis bartensteini*, *L. nodosa*, *L. eichenbergi*, *Gavelinella barremiana*, *Dentalina oligosteagia*, *D. distinct. gracilis* and *Epistomina sp.*

The top of this series is mainly argillaceous and delivered a marine microfaunal association composed with: *Praehedbergella sigali*, *P. primare*, *P. aptiana*, *P. globulifera*, *Globigerinelloides duboisii*, *G. blowi*, *G. solida*, *Leupoldina cabri*, with benthic fauna: *Praedorothia hechti*, *Lenticulina ouachensis*, *L. nodosa*, *L. guttata*, *Gavelinella barremiana*, *Ammodiscus sp* and *Vaginulina robusta* with ostracods: *Cythereis sp.*, *C. arabica*, *C. btaterensis*, *C. geometrica*, *C. laqueiformis*, *Prothocythere triplicata*, *P. algeriana* and *P. bedoulensis*, and the ammonite *Deshayesites deshayesi* marking the Late Bedoulian (Fig. 5).

The biostratigraphic study of the Jebel Oust Barremian–Aptian series allows identifying two sets of black shale. The first one occurred at Early Late Barremian (Fig. 5), and is framed by two biohorizons: *P. sigali* and *G. kugleri* at the base and *H. caillaudi* at the top. It settled at the end of the Early Barremian just before the deposition of the regional landmark formation, with the hard nodular pure limestones that is attributed to the Late Barremian (Figs. 2 and 5). We date this event in Jebel Oust "Early Late Barremian" but we must adopt the same designation "MBE: Middle Barremian Event" establish for the northern Tethyan margin event (Coccioni et al. 1992, 2003, 2006; Sprovieri et al. 2006; Bodin et al. 2009; Malkoc and Mutterlose 2010;

Yilmaz et al. 2012). That's why this first set of black shale is considered to have a Middle Barremian age that had been formed when a short anoxic/dyoxic event occurred. It is the first recorded evidence of Mid-Barremian Event (MBE) in the Southern Tethyan margin that is widely represented along the Northern margin (Coccioni et al. 2003; Yilmaz et al. 2012; Wulf et al. 2020). The second set of black shale is dated by Ear Aptian (Figs. 2 and 5) and was settled between two biohorizons. The basal one is marked by *G. blowi* and *Deshayesites deshayesi* biozones, and the upper one is formed by *L. cabri*. This event is taken in the "Selli Event" OAE1a widely extended to the Tunisian furrow domain (Talbi 1991; Soua 2016; Talbi et al. 2018) as well as to the north Tethyan margin (Bralower et al. 1994; Leckie et al. 2002; Luciani et al. 2006; Graziano 2013; Moosavizadeh et al. 2014).

Distribution of organic matter in various sedimentary facies

The use of the basic Rock–Eval pyrolysis parameters is to identify the petroleum source rock potential. The results of rock samples pyrolysis of Barremian–Aptian series are given in Tables 1, 2, 3, 4, 5, and 6. They indicate low to high TOC contents ranging from 0.06 and 0.93% weight in the Barremian series with an average of about 0.5% weight (Table 1). Those of the Early Aptian facies record TOC values ranging from 0.27 to 2.49% with an average of 1.28% weight (Table 2).

Table 1 Organic geochemical data from Rock–Eval pyrolysis: total organic carbon (TOC), hydrogen index (HI), oxygen index (OI) and anoxic index (HI/OI) of Jebel Oust Barremian series

Age	Sample	TOC (%)	HI (mg/g)	OI (mg/g)	HI/OI
Barremian	OAH14	0.54	115	339	0.33
	OAH12	0.06	433	17	–
	OAH10	0.16	769	425	1.81
	OAH09	0.49	82	112	0.74
	OAH07	0.82	87	71	1.22
	OAH05	0.93	86	85	1.01
	OAH03	0.60	110	282	0.39
	OAH02	0.47	123	149	0.83
	OAH01	0.33	112	406	0.28
	Average	0.49	213	209	0.82

Table 2 Organic geochemical data from Rock–Eval pyrolysis: total organic carbon (TOC), hydrogen index (HI), oxygen index (OI) and anoxic index (HI/OI) of Jebel Oust Aptian series

Age	Sample	TOC (%)	HI (mg/g)	OI (mg/g)	HI/OI
Aptian	OAH34	0.27	96	200	0.48
	OAH33	0.47	115	143	0.80
	OAH29	2.49	174	45	3.86
	OAH28	2.38	135	63	2.14
	OAH25	1.53	132	142	0.93
	OAH22	0.57	128	98	1.30
	Average	1.28	130	115	1.13

Table 3 Average organic geochemical data recorded in the various sedimentary facies of Jebel Oust Barremian–Aptian series

Sedimentary facies	TOC (%)	HI (mg/g)	OI (mg/g)	HI/OI
Black shales	1.66	120	66	2
Marls	0.76	117	292	0.4
Limestones and silicified black shales	0.35	302	195	1.5

Table 4 Average organic geochemical data from Rock Eval analyses recorded in MBE and OAH1a organic-rich black shales of Jebel Oust

Anoxic event	Sample	TOC (%)	Tmax (°C)	TOC ₀ (%)	S ₂ ₀ (mg)	TR (%)	RHC (mg/g)	RHC (%)	EHC (%)
OAE1a	OAH29	2.49	447	4.35	19.88	63	4.78	24	76
	OAH28	2.38	448	4.12	18.83	72	3.55	19	81
	Average	2.43	447	4.23	19.35	67	4.16	21	78
MBE	OAH07	0.82	453	1.43	6.53	82	0.84	13	87
	OAH05	0.93	455	1.62	7.4	82	0.85	18	82
	Average	0.87	454	1.52	6.96	82	0.84	15	84

Table 5 Contents of present day and initial total organic carbon (TOC, COT₀), initial hydrocarbon generation potential (S₂₀), transformation rate (TR), retained hydrocarbons (RHC), expelled hydrocarbons (EHC), hydrocarbon generation potential, oil saturation index (OSI) and T_{max} in the Jebel Oust Barremian series

Age	Sample	TOC (%)	TOC ₀ (%)	S ₂ ₀ (mg/g)	TR (%)	RHC (mg/g)	RHC (%)	EHC (%)	HGP (mg/g)	OSI	Tmax (°C)
Barremian	OAH14	0.54	0.75	1.60	54	0.67	42	58	0.62	9	–
	OAH12	0.06	0.09	0.57	–	0.29	51	49	0.26	50	428
	OAH10	0.16	0.25	1.59	–	1.45	91	9	1.23	137	460
	OAH09	0.49	0.68	1.45	67	0.45	31	69	0.40	10	445
	OAH07	0.82	1.43	6.53	82	0.84	13	87	0.71	16	453
	OAH05	0.93	1.62	7.4	82	0.85	18	82	0.80	5	455
	OAH03	0.6	0.84	2	56	0.72	36	64	0.66	10	422
	OAH02	0.47	0.74	4.70	66	0.63	13	87	0.58	11	449
	OAH01	0.33	0.46	0.98	55	0.41	42	58	0.37	12	–
	Average	0.49	0.76	2.98	66	0.70	37	62	0.62	29	444

Table 6 Contents of present day and initial total organic carbon (TOC, COT₀), initial hydrocarbon generation potential (S₂₀), transformation rate (TR), retained hydrocarbons (RHC), expelled hydrocarbons (EHC), hydrocarbon generation potential, oil saturation index (OSI) and T_{max} in the Jebel Oust Aptian series

Age	Sample	TOC (%)	TOC ₀ (%)	S ₂ ₀ (mg/g)	TR (%)	RHC (mg/g)	RHC (%)	EHC (%)	HGP	OSI	Tmax (°C)
Aptian	OAH34	0.27	0.43	2.70	73	0.30	11	89	0.26	15	447
	OAH33	0.47	0.74	4.70	68	0.59	13	87	0.54	11	448
	OAH29	2.49	4.35	19.88	63	4.78	24	76	4.33	18	446
	OAH28	2.38	4.12	18.83	72	3.55	19	81	3.21	14	444
	OAH25	1.53	2.1	3.80	47	2.09	55	45	2.02	05	439
	OAH22	0.57	0.79	1.69	49	0.86	51	49	0.73	23	451
	Average	1.28	2.09	8.60	62	2.03	29	71	1.84	14	446

Analysis of TOC content according to the lithological variations shows that black shales record the highest content with a mean value of 1.66%. The marls contain moderate TOC values middling 0.76%. While the average TOC content in limestones and silicified black shales are low, not exceeding 0.35% (Table 3).

The evaluation of the quality of the organic matter using the hydrogen (HI) and the oxygen index (OI) as quality and origin kerogen indicators shows (Table 3), first that the HI average values are moderate and similar in black shales and marls (respectively equal to 120 and 117 mg HC/g

TOC) but higher in silicified black shales and limestones (302 mg HC/g TOC). Average OI values are low in black shales (66 mg CO₂/g TOC), higher in marls (295 mg CO₂/g TOC) and medium in silicified black shales and limestones (195 mg CO₂/g TOC). These variations indicate that the organic-rich black shales record the best quality of the kerogen because they are with low oxygen compounds representing a very well preservation degree in an anoxic/dyoxic deposited environment. However, kerogen of marls is rich oxygen compounds deriving probably from continental origin (Petersen et al. 2008). Whereas organic-poor facies that

Fig. 7 Diagram HI/OI showing type I (alga/sapropelic), type II (phytoplanktonic) and type III (lignocellulosic) kerogen in the different sedimentary facies from Jebel Oust Barremian–Aptian series

are represented by silicified black shales, and limestones are similar to hydrocarbons that may have migrated from organic-rich facies (organic-rich black shales and organic-rich marl). This is demonstrated by OSI (oil saturation index) values which recorded a high values, greater than 100% (Jarvie 2012) (Fig. 6).

The HI/OI diagram (Fig. 7) can display and compare the origin and quality of organic compounds of the different sedimentary facies. This diagram, established by Espitalié et al. (1977) shows the three lines of the nature and origin of kerogen: type I, type II and type III. These three lines differ in the values of HI and OI. Type I kerogen is characterized by a high initial HI and a low initial OI. This kerogen is derived from organic algal / sapropelic material. Type II kerogen is characterized by intermediate HI and OI values which originate mainly from marine organic matter deposited in reducing sedimentary environments. Finally, type III kerogen has a low initial HI and OI and is of terrestrial plant origin. It shows that black shale organic matter kerogen is marine plankton: type I, II. While marls contain allochthonous organic matter of lignocellulosic origin: type III. Limestone and silicified black shales samples are with intermediate organic matter quality (Fig. 7).

All these results demonstrate that organic-rich black shales can be considered as the real source rock facies

as well as the organic-rich marls. The organic-poor limestones and silicified black shales can be considered as thin reservoirs intercalated within the shale resource. HI/OI index that is equal to S₂/S₃ index (Calvert et al. 1992) characterize the anoxic degree. It is higher in organic-rich black shales (average HI/OI = 2) than in organic-rich marls (average HI/OI = 1.5). Black shales are then the most representative facies of anoxic events. They can be considered as the indicators facies of these events which contain the highest organic matter contents that are preserved under dyoxic/anoxic conditions. Through their millimeter laminations, they would have recorded the seasonal variations, the extreme seafloor stability and the hydrodynamic low-energy provided by water-mass stratification (Demaision and Moore 1980).

Characterization of the two oceanic anoxic events MBE and OAE1a

MBE anoxic event that marks the Early Barremian (Figs. 2 and 5) is represented by six layers laminated black shales alternating with thin dark-clay limestones and marls (Fig. 8). These black shales recorded a high TOC content with an average closing to 0.9% rock (Table 3). The second event OAE1a dated Early Aptian (Figs. 2 and 5) is represented by two layers of black shale enclosed in dark-gray marls. It recorded the highest TOC values with an average nearby 2.5% (Table 4) (Fig. 8). Hydrogen index values are moderate prone hydrocarbons in the two anoxic events (<300 mg/g TOC) caused by the relatively high maturity degree (oil window) and hydrocarbons generation and expelledation that is demonstrated below.

The HI/OI ratio which is equal to the S₂/S₃ indicates the degree of anoxia of the deposition environment (Calvert et al. 1992, Talbi et al. 2018). The highest values of this ratio are recorded in the Early Aptian (HI/OI > 3) (Table 2), while Barremian interval is marked by a moderate HI/OI ratio (1 < HI/OI < 2) (Table 1). Figure 8 shows multiple peaks in HI/OI curve but two peaks are appropriating to anoxic conditions because they are related to the high organic matter contents (the other small peaks are not significant because of their low TOC contents). These two peaks related to the high anoxic degree correspond to the two Barremian–Aptian anoxic events. The highest peak gives the undisputed supremacy of OAE1a anoxic crisis compared to MBE one (Fig. 8). We consider that the dyoxic/anoxic conditions that were developed at the Early Aptian would have been initiated at the Early Late Barremian event and had reached the highest level when the OAE1a occurred. This may be related to the several modifications in biotic and abiotic factors that could have caused

Fig. 8 MBE and OAE1a oceanic anoxic events correlation with the organic geochemical parameters (TOC, HI, OI and HI/OI) of Jebel Oust Barremian–Aptian source rocks compared to their Western

North Atlantic equivalents (Leckie et al. 2002) and the Vail sea level curve (Vail et al. 1977)

by the global Tethyan changes (Coccioni et al. 2003, Huck et al. (2013); Yilmaz et al. 2012, Wulf et al. 2020).

According to several authors, MBE is an exceptional event associated with large-scale changes in the ocean-climate system related to the rapid ocean formation of the Ontong-Java Plateau, which ultimately led to OAE1a. The MBE coincides with a series of biotic and geochemical events occurring before OAE1a and could be considered as a real shift in the long-term Barremian–Aptian cycle of black shale deposit and evolutionary changes in several fossil groups (Coccioni et al. 2003, Yilmaz et al. 2012, Wulf et al. 2020).

Estimated primary organic content and unconventional oil richness

Rock Eval parameters as common geochemical measurements such as total organic carbon (TOC), hydrocarbon generation potential (S_2) and free hydrocarbons (S_1) are present day values. These measurements are carried on rocks whose organic material have undergone a less or high thermal maturity degree and therefore lacked a part of their organic compounds (hydrocarbons) by migration. The initial state in which the organic matter was immature that can be estimated by calculating the initial organic parameters. So, a method has been defined by Jarvie et al. (2007) that allows

to determine the transformation ratio (TR), the initial total organic carbon (COT_0) and the initial hydrocarbon generation potential (S_{20}) knowing the initial hydrogen index (IH_0) (Jarvie et al. 2007). The transformation ratio (TR) represents the percentage of kerogen having undergone thermal cracking between the initial state (state of immature organic matter) and the current state (present day measures):

$$TR = \frac{HI_0}{HI} \times 100$$

To apply this method for evaluating hydrocarbon generative capacities, IH_0 is estimated at 475 for black shales with type I, II organic matter, and at 250 for marls with type III organic matter. For organic-poor facies (limestones and silicified black shales), we use the mean of the two values (Jarvie et al. 2007). The estimated values of TOC_0 , S_{20} and TR are given in Tables 4, 5, and 6 and Fig. 9. TR is about 50% in the marl organic matter, and ranges from 60 to 80% in black shales. This difference reflects the diversity in organic matter origin in these two types of facies, which is type I, II in black shales and type III in marls. In fact, type III organic matter is more rich in aromatic compounds and provides more energy to crack its hydrocarbons. The initial hydrocarbons generation potential (S_{20}) are exceptionally higher in black shales particularly those recorded in OAE1a than in the other sedimentary facies. They range from 18.83 to 19.88 mg HC/g TOC and from 6.53 to 7.40 mg HC/g TOC, respectively, in OAE1a and MBE organic-rich black

Fig. 9 Variations of initial OM contents (TOC_0 , $S2_0$) transformation ratio (TR), retained and expelled hydrocarbons (RHC, EHC) of Jebel Oust Barremian–Aptian series

shales. In marls, $S2_0$ is with relatively lower to moderate values (0.98–3.8 mg/g) (Table 4) (Fig. 9). This could be explained by a greater degree of preservation and/or organic productivity in the depositional environment of the black shales than in those of marls. However, organic-poor limestones and silicified black shales have recorded the lowest values of TOC_0 , $S2_0$ and TR. Those sedimentary facies represented a short oxic phase or resedimented deposits that may be moved throw sliding down slopes.

It should be noted that $S2_0$ includes retained (RHC: $S1 + S2$) and expelled hydrocarbons (EHC). Tables 5 and 6 show that RHC, which are in place unconventional hydrocarbons vary from 0.3 to 1.45 and from 0.3 to 4.78 mg/g rock and represent about 37% and 29% of $S2_0$, respectively, in the Barremian and the Early Aptian series. Average EHC is twice greater than RHC and is, respectively, represented by 63 and 71% of $S2_0$. This demonstrates release and migration of hydrocarbons outside the organic-rich sedimentary facies then the relatively high maturity degree that is in oil window stage. The oil saturation index (OSI) is ranging from 5 to 137 (Tables 5 and 6) and is the witness of hydrocarbons migration (Fig. 6). Eventually, hydrocarbon generation potential (present day values) is still relatively higher in spite of relatively high maturity degree. Kerogen has not yet crushed its full potential for hydrocarbon production. The positive correlation between HGP and the TOC contents is an evidence of the source rock capacity to generate hydrocarbons in future and that's

why it is considered such a good oil prone unconventional resource system.

Figures 6, 8, and 9 show two peaks that correlate positively with the two oceanic anoxic events: OAE1a and MBE. The positive correlation between TOC_0 , $S2_0$, HGP, TR, RHC, EHC and TOC reveals the existence of two main source rocks with moderate to important unconventional shale oil reserves. These source rocks are not homogenous and contain sedimentary intercalation of organic-rich and organic-poor facies. The first ones constitute the real source rock facies, and the second ones can be considered reservoir thin rocks. The little organic matter contained in the latter appears to result from the hydrocarbons migration produced and released by the organic-rich facies. The OSI that exceeds 100% is the proof (Jarvie 2012, 2015) (Tables 5 and 6).

Shale oil resource system characterization

The petroleum system elements consist of a mature source rock, migration pathway, reservoir rock and trap (Al-Hajeri et al. 2009). In the unconventional resource systems, all these components are associated in the same rock. The studied Barremian–Aptian series includes three types of facies: organic-rich black shales, organic-rich marls and organic-poor limestones and silicified black shales. The first two types constitute source rocks with the difference that black shales record the highest organic matter contents. They are good to very good hydrocarbon prone facies with an

excellent organic matter quality which was very well preserved in the sedimentary environment. Organic-rich marls constitute a source rock of moderate quality. Its organic matter is lignocellulosic that may be carried with detrital particles. The organic-poor limestones can play the role of thin hydrocarbon reservoir and trap. Oil crossover constitutes an undeniable proof for that (Fig. 6). Since shale resource is made up of intercalated organic-rich and organic-poor sedimentary facies, when the mature stage occurs under high temperature and pressure, oil and gas saturation is attempted by thermal cracking kerogen which can trigger horizontal migration. As hydrocarbon generation and migration proceed with maturity progress, vertical migration takes place and oil leaves organic-rich facies (black shales) with high geochemical confinement degree to lodge in the poor-organic facies (limestones) that constitute close thin hydrocarbon reservoirs. This phenomenon was well demonstrated by geochemical and petrographical indicators for the Aptian-Albian shale resource system outcropping in the same region (Talbi et al. 2018). Those organic-poor facies may be attractive for unconventional oil explorers because they may help the hydrocarbons drainage by hydraulic fracturing operations due to the low degree of geochemical confinement (Jarvie 2012). Another element of the shale resource system is the open fractures that are frequent across the series and indeed, lead to hydrocarbon migration and drainage. Depending on the geologic and geochemical analysis and the degree of thermal maturity that is liquid oil window (Fig. 6), the two source rocks that had deposited during the two oceanic anoxic events OAE1a and MBE can be considered regarding Jarvie (2012) classification as "shale oil hybrid systems with a combination of juxtaposed organic-rich and organic-lean facies associated with open fractures".

Factors controlling source rock deposition

The Middel Cretaceous oceanic anoxic events had recorded major black shales that provided most of the world's fossil fuels (Jenkyns 1980; Arthur et al. 1990; Ghasemi-Nejad 2009; Al-Ameri and Al-Obaydi 2011; Farzadi 2006; Sadooni and Alsharhan 2003; Genik 1993). Early Cretaceous anoxic events appear as preludes to them (Coccioni et al. 2003; Huck et al. 2013). The current knowledge strongly suggests that the black shale deposits were associated with climatic variations leading to changes in surface water and deep water ecosystems (Leckie et al. 2002). This is the case of the MBE, whose hot humid climate ensured intense organic productivity and profound biotic changes (Malkoc and Mutterlose 2010; Ohkouchi et al. 2015) that could have probably primed the OAE1a (Bastide 2014; Huck et al. 2011). Marine productivity favored by increased nutrient supply and organic matter (OM) preservation was the effect of anoxic/dysoxic conditions (Leckie et al. 2002). Those are the

major processes used to explain the deposition of organic-rich black shales. Depletion of oxygen in the substrate and even in the water column causes incomplete degradation of organic matter by anaerobic bacteria, leading to high TOC enrichment in the sediments (Leckie et al. 2002). In the Barremian–Aptian outcrop series of Jebel Oust, black shales generally exhibit good conservation of type I and type II organic matter. Therefore, marls preserve less TOC contents than black shales. Its organic matter content is made up of continental lignocellulosic origin which explains the higher OI values (OM type III). Then black shales are better source rocks than marls. Organic-poor facies are represented by limestones interbedded with marls and black shales and appear playing the role of thin hydrocarbon reservoirs. In fact, OSI is higher in limestone than in organic-rich black shales and marls. This variation in organic matter richness and quality in the different facies seems due to the regional and local geodynamic and paleogeographic evolution that impact the basin filling history (Liang et al. 2017; Talbi et al. 2018; Han et al. 2019). Two transgressive–regressive cycles bounded by two organic-rich black shales are deposited at the Jebel Oust region that was an intracratonic basin formed by synjurassic rifting (Castany 1954; Chihi et al. 1984; Ben Ayed 1986) but whose collapse would certainly have been accelerated during the Early Cretaceous (Jauzein 1957; Ferjani et al. 2018). This basin was structured in horst and graben and tilted blocks, resulting from North to Northeast-trending regional extension (Fig. 3) developed along transforming EW faults (Ben Ayed 1986; Perthuisot et al. 1988; Naji et al. 2018). Since extensional rift tectonic origin was created, instability of the bottom sea that prevents water stratification at the start of each sedimentary cycle, marl sediment production dominates the basin filling history at the beginning of the sedimentary cycle. The tectonic instability may induce low accumulation and preservation of OM in the basin that filled with organic and inorganic clastic debris from various sources. This is the case of marls which are associated with allochthonous OM with continental origin. The marls, which are filling depressions, show variations in thickness throughout the region. Above these marls, the black shales are generally deposited with a reduced and almost invariable thickness. The very fine regular laminations which characterize black shale deposits suppose a regular and extremely stable bottom (Talbi et al. 2018). The quantities of organic matter often well preserved in black shales assume the existence of stratified water bodies and a minimum oxygen bottom at the water–sediment interface. Then, black shale formation needs extreme tectonic stability, low-rate deposition and weak hydrodynamic interaction (Knapp et al. 2017) that favor their widespread deposition covering the major part of the tectonic deformation within the basin (Fig. 3).

Each sedimentary cycle including the three stages (Fig. 3): basin structuring, basin marly filling and finally black shales deposition occurred during the transgression, at the maximum flooding surface (Vail et al. 1977; Jacquin and De Graciansky 1988; Frimmel et al. 2004). This is the case of OAE1a and MBE as shown in Fig. 8. Then, black shales will be covered by oxic deposits (organic-poor limestones) before the initiation of the next sedimentary cycle. Indeed, the Early Late Barremian interval with poor-organic siliceous black shales and pure white nodular limestone overlie the organic-rich black shales of the MBE. The white nodular limestone shows gravity-driven deformation structures of the soft-sediment and abundant slumps at submarine slopes induced by North to Northeast-trending extension deformations (Naji et al. 2018). Those seafloor deformations accelerate the Late Barremian–Early Aptian sedimentary cycle in which OAE1a took place.

Hydrocarbons may be migrated using some of the pre-existing reactivation normal faults. Retained hydrocarbons would be accumulated in subtle traps due to the presence of rock heterogeneities. This phenomenon was already well analyzed and demonstrated for organic-rich black shales of the Aptian-Albian series (Talbi et al. 2018). Then petroleum system related to the Barremian–Aptian interval of Jebel Oust series according to the Jarvie (2012; 2015) classification is assimilated to **"shale oil hybrid systems with a combination of juxtaposed organic-rich and organic-lean facies associated with open fractures"**.

Conclusion

The organic-rich black shales deposited in the Jebel Oust region are found within marine marly series containing rich microfauna with representative planktic benthic foraminifera assemblages. The first organic-rich black shale level, as recorded in the southern Tethyan margin, is dated Early Late Barremian and coincides with the occurrence of MBE which is widely represented in the northern Tethyan margin and seems to be the prelude of the second organic-rich black shale deposit that is dated Early Aptian and coincides with the OAE1a. Two transgressive-regressive cycles were deposited during the Early Cretaceous (Barremian–Aptian interval). They are mainly represented by marls with intercalations of limestones and black shales. Rock–Eval pyrolysis data distinguish black shales by means of high organic matter (OM) contents, marls with moderately rich OM and silicified black shales and limestones as organic-poor facies. Average present day total organic carbon (TOC) contents are, respectively: 1.66, 0.76 and 0.35% wt. Type I and type II marine organic matter characterize only black shales with low hydrogen and oxygen index values (average HI = 120 and OI = 66) due to the relatively high maturity degree that

corresponds to the end of the liquid oil window, whereas type III continental OM characterizes marls with high OI and low HI values (average HI = 117 and OI = 292) and is interpreted as a contribution of an organic detrital sediments. Limestones recorded dispersed HI and OI values (average HI = 117 and OI = 121). Their OM is identified as hydrocarbons that would have migrated from source rocks (black shales) and trapped in the porosity of these organic-poor facies that constitute a thin reservoir rock intercalated with organic-rich facies. Then organic-rich black shales could be considered as dyoxic/anoxic facies and as oil prone source rock.

Since the sedimentary basin being of tectonic origin, geodynamic had controlled the organization of the fill basin system. The basin that was structured in horst and graben and tilted blocks, resulting from North to Northeast-trending regional extension, developed along transforming EW faults, got filled up early by marls that were deposited in the depressions formed after each distensive tectonic activity. Parallel to the gradual change in the mean sea level, the unevenness of the seafloor inequality gradually fades and the organic-rich black shales settle coinciding with the maximum flooding events of marine transgressions and the start. MBE black shales recorded 1.5% wt for average TOC₀ and nearby 7 mg HC/g for S₂₀.

The higher values of estimated TOC₀ (4% wt) and S₂₀ (19.36 mg HC/g) characterize OAE1a black shales. This suggests that OAE1a represents an increase in marine organic productivity and / or environmental proxies indicating anoxia-dysoxia that may be initiated at MBE.

Present day OM contents recorded in OAE1a black shales are higher than in those of MBE. TR is more important for MBE black shales (82%) than for OAE1a ones (67%) due to the high maturity degree for the Barremian sediments. In consequence, the major part of the initial hydrocarbons is crashed and expelled from the two source rocks. Average RHC is nearby 20%. However average EHC is about 80%. OSI is with high dispersed values (9–137) which indicate hydrocarbon released and expelled from source rocks. As long as those source rocks present significant quantities of hydrocarbons in place that can circulate since the degree of thermal maturity corresponds to the oil window, these two source rocks are considered to be an unconventional shale oil system. Taking into account the tectonic structure, the geochemical parameters and the various sedimentary facies, the shale resource system is mainly represented by black shales formed during the two anoxic oceanic events MBE and OAE1a which are oil prone source rocks categorized as **"shale oil hybrid system with a combination of juxtaposed organic-rich and organic-lean facies associated with open fractures"**.

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Declarations

Conflict of interest The authors declare that they have no conflicts of interest.

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