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# Formation of microbial organic carbonates during the Late Jurassic from the Northern Tethys (Amu Darya Basin, Uzbekistan): Implications for Jurassic anoxic events

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## ABSTRACT

The Late Jurassic was a period of major global carbon cycle perturbations with episodes of anoxia leading to regional accumulation of organic matter in sediments worldwide. The Tubiegatan section (SW Gissar Mountains, Uzbekistan) located in the Northern Tethys, shows atypical organic-rich limestone and marl deposits (up to 6% of total organic carbon) marked by pronounced **negative** excursions of  $\delta^{13}\text{C}_{\text{carb}}$  (amplitude of *ca.* 12‰) and  $\delta^{13}\text{C}_{\text{org}}$  (amplitude of *ca.* 4‰) recorded during the Middle Oxfordian (Transversarium Zone). A transdisciplinary approach including sedimentology, palynofacies characterization, mineralogy, organic and inorganic geochemistry was carried out to elucidate the origin of these organic-rich deposits. Highest TOC are measured in nodular limestones, and lowest  $\delta^{13}\text{C}_{\text{carb}}$  values in thinly laminated facies consisting in alternances of infra-millimeter-thick organic and carbonate laminae. In the latter, the presence of organic-carbonate peloids and of possible remnants of exopolymeric substances associated with clay indicate that these structures are probably mineralized **laminated** benthic microbial mats (i.e., stromatolites). Rock-Eval pyrolysis coupled to palynofacies analyses point to a dominant altered marine organic matter of probable algal/microbial

origin, with subordinate continental phytoclasts inputs in the upper part of the organic-rich interval. Trace elements (U/Th, V/Cr and Mo/Al ratios) indicate two anoxic episodes coinciding with the highest TOC, punctuated by dysoxic periods. Such O<sub>2</sub>-depleted conditions have allowed the preservation and probably the development of anaerobic microbial communities in the microbial mats. In these latter, sulfate reduction probably had a significant contribution to the production of carbonates, which would explain the precipitation of pyrite and the relatively low  $\delta^{13}\text{C}_{\text{carb}}$  values. The progressive decrease then disappearance of kaolinite from the base of the organic-rich interval, is interpreted as a progressive aridification of the Amu Darya Basin during the Transversarium Zone, culminating with the progradation of a large-scale gypsum sabkha overlying the organic deposits. Overall, the organic-rich deposits could record the onset of the disconnection of the Amu Darya Basin from the open sea to the south, induced by compression and subsequent uplifts in the Afghan and Central Iranian blocks. The elevated evaporation, coupled with the presence of hydrological barriers (such as coral reefs) could have led to the formation of local to regional anoxic conditions in the Amu Darya Basin. Similar microbial organic accumulations are recently known throughout the Tethys (e.g., Arabian Plate, Western Europe) and from other oceans (e.g., Central Atlantic, Pacific) during the Late Jurassic, suggesting common controlling factors. The increase of organic matter storage worldwide coupled with potential methane release could have in turn induced major perturbations of the carbon cycle during the Oxfordian-Kimmeridgian interval. The relatively shallow anoxia model proposed in this study contrasts with the well-known organic carbon-rich pelagic models proposed for the Jurassic anoxia (e.g., Toarcian, Kimmeridgian) and Cretaceous OAEs.

**Keywords:** Late Jurassic, Amu Darya Basin, microbial/laminated sediments, organic-rich carbonates, anoxia, stable isotopes

## 1. INTRODUCTION

The Late Jurassic is a period marked by multiple anoxic events (e.g., Trabucho-Alexandre et al., 2012; Martinez & Dera, 2015). More specifically, the Oxfordian and Kimmeridgian Stages are characterized by episodes of regional anoxia resulting in the accumulation and preservation of organic matter (OM)

in several sedimentary formations throughout the world, e.g., the Staffin Shale Fm. (Middle Callovian–Lower Kimmeridgian, Isle of Skye, Scotland; Pearce et al., 2005), the Knollenkalk Fm. (Middle Oxfordian, Jura, Switzerland; Rais et al., 2007), La Manga Fm. (Middle–Upper Oxfordian, Neuquen Basin, Argentina; Palma et al., 2015), the Hanifa Fm. (Oxfordian–Lower Kimmeridgian, Central Arabian Basin, Saudi Arabia; Droste, 1990; Vahrenkamp et al., 2015), the Smackover Fm. (Oxfordian–Kimmeridgian, Gulf Coast, USA; Oehler, 1984), the Spiti Shale Fm. (Oxfordian–Berriasian, Malla Johar, India; Jain et al., 1984), the Akkuyu Fm. (Lower Kimmeridgian–Tithonian, Taurus Mountains, Turkey; Baudin et al., 1999), the Kimmeridge Clay Fm. (Kimmeridgian–Tithonian, Dorset, UK; Tribovillard et al., 1994). A large number of these organic rocks show a fine-grained laminated fabric, sometimes called organic varves. This facies has been interpreted as mineralized microbial mats (or stromatolites) that formed in shallow stratified, hypersaline lagoons (Bernier and Enay, 1972; Tribovillard et al., 2000; Warren, 2011). Warren (2011) postulated that such laminated organic sediments could also reflect pronounced seasonal **salinity and nutrient variations** leading to blooms in primary productivity, which in turn would result in the formation of organic laminae.

At the global scale, the role of climate and tectonic on the multiplication of organic-rich formations during the Late Jurassic is still debated. On the Arabian platform, the Hanifa and Jubaila Fms. have accumulated in partially isolated **and stratified** intrashelf basins. In a recent review, Vahrenkamp et al. (2015) link the formation of such basins to a tectonic uplift at the margin of the Arabian Plate. Based on the presence of biosiliceous deposits, Eltom et al. (2017) have related the elevated organic productivity in these basins to regional upwellings developing at the shelf edge. Both models may explain the formation of organic sediments on large epeiric platforms (Warren, 2011). In addition, several authors proposed that the increase in organic productivity and the multiple anoxia recorded during the Late Jurassic could have been caused by elevated atmospheric  $p\text{CO}_2$  due to enhanced magmatic activity at oceanic ridges and in subduction zones (Jones and Jenkyns, 2001; Dera et al., 2011; Meer et al., 2014; Georgiev et al., 2017).

In the Western Tethyan domain, the Middle–Late Oxfordian interval is characterized by elevated carbon isotope values from bulk carbonate ( $\delta^{13}\text{C}_{\text{carb}}$ ) as reported by several authors (Bartolini et al., 1999; Louis-Schmid et al., 2007; Rais et al., 2007; O’Dogherty et al., 2018), interpreted as the result of

86 palaeoenvironmental events (e.g., major currents reorganization, climate warming, sea-level rise, higher  
87 carbonate accumulation rates). Within this overall positive  $\delta^{13}\text{C}_{\text{carb}}$  interval however, during the  
88 Transversarium Zone, a marked  $\delta^{13}\text{C}_{\text{carb}}$  negative excursion was reported in the Jura and the Subalpine  
89 Basin by Padden et al. (2001). A less well-dated negative excursion was also documented in the whole  
90 Western Tethyan domain at the Middle-Late Oxfordian transition both in shallow water fossils (e.g.,  
91 bivalves) and in deeper benthic organisms (e.g., belemnites; Martinez and Dera, 2015). By contrast, no  
92 well-dated  $\delta^{13}\text{C}$  record is available for this period in the Northern Tethys.

93 The Amu Darya Basin (ADB) is a vast sedimentary basin (*ca.* 400,000 km<sup>2</sup>) of the Northern Tethys.  
94 This basin hosts the organic-rich Khodjaipak Formation, which is a major source rock for hydrocarbons  
95 (Klett et al., 2006, 2012). This formation is dated from Late Oxfordian to Kimmeridgian in age  
96 (Abdullaev, 2004 or Mordvintsev, 2015). These 10 to 30 m thick deposits have been identified in more  
97 than two hundreds boreholes on the northeastern margin of the ADB (Besnosov and Mitta, 1995;  
98 Abdullaev et al., 2010), but outcrop only at two locations in the south-west Gissar mountain range (SW  
99 Gissar). In this study, field sedimentological investigations were carried out in one of these localities  
100 (Tubiegatan, Uzbekistan). To date, no detailed sedimentological, mineralogical or geochemical study  
101 has been published on these deposits. The Khodjaipak Fm. is located near the top of the Upper Jurassic  
102 carbonates of the Kugitang Series, and could have accumulated in depressions surrounding reefal  
103 buildups formed on the contemporaneous carbonate platforms (Mirkamalov et al., 2005). However, the  
104 absence of outcrops between the organic deposits and their potential stratigraphic counterparts in the  
105 SW Gissar implies a re-evaluation of the platform to basin correlations. The presence of ammonites in  
106 the Tubiegatan section however allows herein to specify the timing of the main palaeoenvironmental  
107 events occurring at this period on the northern margin of the ADB.

108 In this study, the Khodjaipak Fm. organic deposits are characterized through a transdisciplinary  
109 approach including sedimentology, biostratigraphy, petrography, clay mineralogy, and organic and  
110 inorganic geochemistry. Our main objectives are to (i) determine the composition and the mode of  
111 formation of such organic deposits, (ii) propose a high resolution biostratigraphic and carbon isotope  
112 stratigraphic framework for the Tubiegatan section and (iii) establish the stratigraphic relationships with  
113 the contemporaneous carbonate platform in order to specify the depositional conditions of the

Khodjaipak Fm. The resulting model will be compared to similar Upper Jurassic deposits worldwide in order to determine potential common patterns in their formation and the potential impact on carbon cycle of multiple organic carbon accumulations.

## 2. GEOLOGICAL SETTING

The Kugitang Series is a thick carbonate platform succession (~100–600 m) deposited on the NE margins of the ADB (**Fig. 1A**) from the Early Callovian up to the Kimmeridgian (Mirkamalov et al., 2005; Nugmanov, 2010; Carmeille et al., 2018). This series crops out in the southwestern Gissar mountain range (SW Gissar), in the northeastern part of the ADB. The Kugitang carbonate platform is subdivided into two major depositional sequences: (i) the Callovian Sequence (CS) equivalent to the Lower Kugitang Series and (ii) the Upper Jurassic Sequence (UJS) equivalent to the Upper Kugitang series (**Fig. 2**; Carmeille et al., 2018). The CS deposition begun in the Early Callovian and lasted at least until the top of the Middle Callovian (Coronatum Zone), or possibly until the Late Callovian (**Fig. 2**; Mitta, 1992). At this time, the SW Gissar was covered by a carbonate ramp. The CS ended with a major subaerial exposure surface probably associated with a hiatus. The UJS records a major reorganization of the platform. A vast low energy lagoon developed, probably protected by a barrier reef outcropping in the Kugitang area (southern part of the SW Gissar; **Fig. 1A,B**). The organic-rich deposits of the Khodjaipak Fm. could be partly time-equivalent of this barrier reef (Abdullaev, 2004). The Khodjaipak Fm. dated as Upper Oxfordian and Kimmeridgian (Abdullaev, 1999, 2004; Abdullaev and Mirkamalov, 2001) is considered as a major type-II kerogen source rock (Ulmishek, 2004). On the Chardzhou step (NE margin of the ADB; **Fig. 1A**), more than two hundred boreholes from major gas fields have drilled this formation (e.g., North Kamashi, South Alan, Ayzovat 7; Ulmishek, 2004; Mirkamalov et al., 2005; Abdullaev et al., 2010; Mordvintsev, 2015).

The Khodjaipak Fm. crops out only in two localities close to the Turkmenistan-Uzbekistan border: Tubiegatan and Gaurdak (Besnosov & Mitta, 1995; **Fig. 1B**). We did not obtain authorizations to work on the Gaurdak section, located on the Turkmen side. The focus of this study is the Tubiegatan section, located on the Uzbek side. The section exposes a 30-meter-thick limestone interval belonging to the Kugitang Series (**Fig. 2**), overlain by 10 meters of dark limestone and marl alternation (**Fig. 1C**). The

studied deposits crops out along a *ca.* 1 km wide window on the flank of an anticline, thanks to a local gliding of the **overlying Irgayli Fm.** gypsum (**Figs 1D and 2**).

### **3. MATERIALS AND METHODS**

#### **3.1. Ammonite biostratigraphy and sequence stratigraphy**

A total of 14 ammonites (entire or fragments) were collected **along the measured section**. Six of them were determined to the species level by one of us (RE) using the standard biozonation of the NW Tethyan domain (Cariou and Hantzpergue, 1997). A sequence stratigraphic scheme was built based on Maximum Flooding Surfaces (MFSS) and Maximum Regressive Surfaces or Sequence Boundaries (SBs), following the terminology of Catuneanu et al. (2011).

#### **3.2. Petrography**

A total of 90 samples were collected. To obtain a high-resolution record, one sample was taken every 20 cm on average within the organic-rich carbonates and marls deposits. In the limestone interval (Kugitang Series), we took one sample every 1 m on average. Thin sections were prepared (polished with 0.25  $\mu\text{m}$  alumina powder and impregnated with blue epoxy resin) and observed under a Nikon Eclipse Ci polarizing microscope for 50 samples allowing the characterization of microfacies. Three thin sections were examined with a FEI Quanta 250 FEG environmental scanning electron microscope, under low vacuum. One third of each thin section was etched in 10% acetic acid for 1 minute, in order to remove carbonates and have a better observation of the OM. X-ray spectral microanalyses and X-ray maps were performed using energy dispersive spectrometry (EDS, EDAX Apollo XL) at the I2M laboratory, Bordeaux, France.

#### **3.3. Palynofacies analyses**

Determination of palynofacies have been realized on 28 samples. One sample (12-E1) was almost barren of OM and thus unsuitable for palynofacies examination. The carbonate minerals were first dissolved using hydrochloric acid (HCl). Then, the residues were treated with hydrofluoric acid (HF) in order to eliminate the silicates. For each sample, two aliquots of the remaining organic residues were used to

make a non-filtered slide and a filtered slide using a 10  $\mu\text{m}$  sieve mesh. The slides were then examined under an Axioplan2 Imaging Zeiss microscope. Both qualitative and quantitative observations were made. We used a simplified version of the palynofacies classifications proposed by Whitaker et al. (1992) and Tyson (2012), with some modifications by McArthur et al. (2016). The relative surface area of each classes was estimated on the filtered slides, as they allow a better observation of the relative proportions of phytoclasts and palynomorphs. Further details regarding the palynofacies method used in this study can be found in Schnyder et al. (2017). These analyses were conducted at the Institut des Sciences de la Terre de Paris (ISTeP) laboratory (Sorbonne-Université, Paris, France).

### **3.4. X-ray diffraction**

X-ray diffraction analyses were performed at the Biogéosciences laboratory (Université Bourgogne Franche-Comté, Dijon, France) to identify and semi-quantify mineralogy of the bulk-rock (85 samples) and the  $<2\mu\text{m}$  clay-size fraction (80 samples). Clay minerals were identified in oriented mounts of non-calcareous clay-size fraction, following the analytical procedure of Moore and Reynolds (1989). After removing carbonate (using 2N HCl for marl and 12N HCl for limestone), clays were deflocculated by successive washing with distilled water. Particles finer than 2  $\mu\text{m}$  were concentrated by centrifugation. Diffractograms were obtained using a Bruker D4 Endeavor diffractometer with  $\text{CuK}\alpha$  radiation, LynxEye detector and Ni filter, under 40 kV voltage and 25 mA intensity. Three XRD runs were performed, after air drying (AD), ethylene-glycol solvation (EG), and heating at 490 °C for 2 h. The goniometer performed a scan from 2.5° to 28.5° 2 $\theta$  for each run. Identification and semi-quantitative estimates of clay mineralogy were made according to the position and the area of the (001) basal reflections on the three XRD pattern (Moore and Reynolds, 1989). The identified clay minerals are R1 type illite/smectite mixed-layers (I/S-R1), illite, kaolinite and chlorite. Deconvolution of peak areas of kaolinite ( $d_{002}$ ) and chlorite ( $d_{004}$ ) was performed to discriminate both minerals using MacDiff 4.2.5 software (Petschick, 2001). Other non-clay minerals found in the bulk rock are calcite, quartz, feldspars and pyrite. Semi-quantification of these phases are performed considering mean reflexions (100% intensity) of each phase and using the % $\text{CaCO}_3$  measured by spectrometry.



### 3.5. Rock-Eval pyrolysis

A total of 61 samples were analyzed by Rock-Eval pyrolysis using a Rock-Eval 6 Turbo (Vinci Technologies) at the Institut des Sciences de la Terre de Paris (ISTeP) laboratory (Sorbonne-Université, Paris, France). The following parameters were obtained (Espitalié et al., 1985; Behar et al., 2001): (i) the total organic carbon (TOC, in wt %); (ii) the hydrogen index (HI, in mgHC/g-TOC), calculated from the quantity of pyrolysable organic compounds from S2 peak released during pyrolysis, normalized to TOC values; (iii) the oxygen index (OI, in mgCO<sub>2</sub>/g-TOC), obtained from the amounts of CO and CO<sub>2</sub> from S3 peak released during pyrolysis, normalized to TOC values, and (iv) the T<sub>max</sub>, corresponding to the temperature recorded in the oven at the peak of hydrocarbon production during pyrolysis. Analytical precision is ± 0.1% for TOC, ± 10 mgHC/g-TOC for HI, ± 5 mg CO<sub>2</sub>/g-TOC for OI and ± 2°C for T<sub>max</sub>.

### 3.6. Stable isotope geochemistry ( $\delta^{13}\text{C}_{\text{carb}}$ , $\delta^{18}\text{O}_{\text{carb}}$ , $\delta^{13}\text{C}_{\text{org}}$ , $\delta^{15}\text{N}_{\text{tot}}$ )

The  $\delta^{13}\text{C}_{\text{carb}}$  and the  $\delta^{18}\text{O}_{\text{carb}}$  (bulk carbonate fraction) were measured on 72 samples at the Biogéosciences laboratory (Université Bourgogne Franche-Comté, Dijon, France). About 50-90 µg of powder were produced by using a micro-drill to avoid crack fillings or fossils. Powder was oven-dried for 12 h at 50 °C then weighed with a Mettler Toledo XS3DU ultra-balance (30 to 60 µg, depending on %CaCO<sub>3</sub>). A Kiel IV carbonate preparation device, paired with a Delta V Plus (ThermoFisher) mass spectrometer was used for isotopic analyses. Samples were dissolved at 70 °C by orthophosphoric acid, and isotopic values were calibrated by NBS-19 and Merck standard. Carbon isotopic composition is expressed in per mil (‰) V-PDB, and oxygen isotopic composition in ‰ SMOW. Replicates (every 7 samples) showed reproducibility of ± 0.07 ‰ for carbon and ± 0.1 ‰ for oxygen.

The  $\delta^{13}\text{C}_{\text{org}}$  was measured on 75 samples and the  $\delta^{15}\text{N}_{\text{tot}}$  was measured on 66 samples at the Biogéosciences laboratory (Université Bourgogne Franche-Comté, Dijon, France). After grinding, the carbonate minerals were dissolved using 6N HCl. The powder was then rinsed with purified water and oven-dried at 50°C before analyses. The  $\delta^{13}\text{C}_{\text{org}}$  and  $\delta^{15}\text{N}_{\text{tot}}$  analyses were performed using a vario MICRO cube (Elementar) elemental analyser, paired to an Isoprime (Elementar) isotope ratio mass spectrometer (EA-IRMS). The values were compared to USGS40 L-Glutamic acid ( $\delta^{13}\text{C} = -26.39 \pm 0.04$  ‰;  $\delta^{15}\text{N} = -4.5 \pm 0.1$  ‰) and IAEA-600 Caffeine ( $\delta^{13}\text{C} = -27.77 \pm 0.04$  ‰;  $\delta^{15}\text{N} = +1.0 \pm 0.2$  ‰)

international standards for  $\delta^{13}\text{C}_{\text{org}}$  and  $\delta^{15}\text{N}_{\text{tot}}$ . The  $\delta^{13}\text{C}_{\text{org}}$  and  $\delta^{15}\text{N}_{\text{tot}}$  values are expressed in ‰ V-PDB and ‰ V-AIR respectively. For each sample, replicates showed reproducibility better than  $\pm 0.3$  ‰ for  $\delta^{13}\text{C}$  and  $\pm 0.5$  ‰ for  $\delta^{15}\text{N}$ .

### 3.7. Major and trace element geochemistry

Multi-elemental analyses were performed using ICP-AES for major elements and ICP-MS for trace elements on 23 samples at the spectrochemical laboratory of the Centre de Recherche Pétrographiques et Géochimiques (CRPG, Vandoeuvre-les-Nancy, France). The samples were prepared by fusion with  $\text{LiBO}_2$  and  $\text{HNO}_3$  dissolution. The average uncertainties are about 1% for major elements, 5% for Cr, U and V, and 10% for Co, Mo, and Ni as checked by international standards (Carignan et al., 2001).

Usually, trace elements are Al-normalized in order to dissociate the detrital and authigenic fractions. However, because of fluctuating Al concentrations (from 0.08 to 5%), the use of fraction in excess was preferred to dissociate these two fractions. As proposed by Brumsack (2006), the detrital fraction  $X_{\text{detritic}}$  has been calculated using the formulae:  $X_{\text{detritic}} = \text{Al}_{\text{sample}} \times (X/\text{Al}_{\text{upper crust}})$ . The values of upper continental crust are from McLennan (2001). Then, the “excess” (non-detrital) fraction has been calculated, using the formulae  $X_{\text{xs}} = X_{\text{sample}} - X_{\text{detritic}}$  for each element, which does not show strong correlations with Al and Ti, considered as elements with a detrital origin (Tribovillard et al., 2006; Collin et al., 2015).

## 4. RESULTS AND INTERPRETATIONS

Based on the vertical facies succession, we defined three lithostratigraphic intervals along the Tubiegatan section: (1) a lower carbonate interval; (2) an organic-rich marl and limestone unit corresponding to the Khodjaipak Fm. and (3) a second carbonate interval at the top. In terms of sequence stratigraphy, the whole succession can be interpreted as a transgressive-regressive third-order sequence with a proposed MFS located *ca.* 20 m from the basis of the section. This sequence can be subdivided into three higher frequency depositional sequences (Tu1, Tu2 and Tu3; **Fig. 3**).

## 4.1. Sedimentology and stratigraphy

### 4.1.1. Lower carbonate interval (0–30 m), Tu1 and Tu2 sequences, base of Tu3 sequence

Mudstones (F2a) and pellet-sponge spicule wackestones (F2b) predominate in the first 25 meters of the section (**Fig. 3**). Such mud and pellet-rich facies are indicative of low energy conditions (Carmeille et al., 2018), and in the absence of storm-deposits they are usually placed below the storm wave base (SWB; Wilson, 1975; Carpentier et al., 2007). The diversified photo-independent benthic associations: sponge spicules, benthic foraminifera, echinoderms and oysters, suggest a well-oxygenated bottom water located below the photic zone. Ammonite imprints were also locally observed at the surface of beds. The micritized ooids (F2e) observed at 14 and 29 m could be exported from the inner platform during shallowing-upward trends. Dolomitized *Thalassinoides* burrowed horizons (**Fig. 4B,C**) reflect the formation of firmgrounds possibly recording a decrease in the sedimentation rate and water depth (Carmeille et al., 2018). Consequently, we placed two 4<sup>th</sup> order sequence boundaries (14 m and 27 m) within these burrowed intervals (**Fig. 3**). The occurrence of *Perisphinctes* (*Kranaosphinctes*) sp. would place the top of Tu1 and the basis of Tu2 in the Oxfordian. The dolomitized *Thalassinoides* burrows at the top of Tu2 (25 to 27 m) are locally filled with diagenetic iron oxides.

The overlying carbonate strata (27 to 30 m) are characterized by serpulids and benthic foraminifera (including Lituolids) packstone (F2c) and *Crescentiella morronensis* and brown foraminifera oncoids pack- to floatstone (F2d). They are placed within the sequence Tu3. The last bed of the lower carbonate interval shows a significant fossil concentration: ammonites, belemnite rostra, oysters and benthic foraminifera (F1d). This fossil association indicates the persistence of a good oxygenation of the whole water column. A hardground develops at the top of this bed, likely reflecting a decreased sedimentation rate. Overall, the lower carbonate interval was mostly deposited below SWB and shows evidence for a well-oxygenated water column.

### 4.1.2. The Khodjaipak Fm. (30–40 m), Tu3 sequence

From 30 to 40 m (**Fig. 3**), the series is dominated by organic-rich marls to siltstones (F1a) and microbial boundstones (F1c), interbedded with black nodular mudstones (F1b) and tempestites (F1d). Millimeter-

laminated dark to brown marls to siltstones (F1a; **Fig. 4D**) containing ammonites and entire bivalves in life position are characterized by TOC values comprised between 0.4% and 4%. The black nodules (F1b) found in these dark to brown marls show the highest TOC values, reaching 6% (**Fig. 4E**). F1c consists of sub-millimetric alternations of carbonate and organic laminae (**Fig. 5A-C**), with TOC values comprised between 0.5% and ~2% (**Fig. 3**). Sponge spicules, benthic (rotaliids: *Lenticulina*, *Rheinholdella*?) and planktonic foraminifera are locally concentrated in thin laminae (**Fig. 5D**). Diagenetic anhydrite pseudomorphs and dolomite rhombs are observed in this facies (**Fig. 5B**). F1d corresponds to dark shell beds (**Fig. 4F**) with moderate TOC (0.8-1.6%). The skeletal elements consist of well-preserved, disarticulated and oriented brachiopod and bivalve shells (e.g., *Trichites*, pectinids, oysters), which are associated with abundant rotaliid foraminifera (identical to those observed in F2c), sponge spicules and rare echinoderm fragments floating in a dark-micritic matrix. The ammonite *Subdiscosphinctes* (*Subdiscophinctes*) cf. *richei* De Riaz /*mindowe* Siemiradzki precisely dates this interval to the Schilli Subzone (Transversarium Zone, Middle Oxfordian; **Fig. 3**). **The suggested depositional environment for these organic carbonates is discussed in details in Section 5.3.1.**

#### 4.1.3. Upper carbonate interval and chicken wire gypsum (40–43m), Tu3 sequence

The section is capped by a 2-m-thick carbonates interval characterized by peloid and fine micritized ooid pack- to grainstone (F3a) and stromatolitic boundstone (F3b). These carbonates are overlain by chicken-wire gypsum, typical of sabkha deposits, whose exact thickness cannot be determined (F4; **Fig. 3**).

#### 4.2. Petrography of microbial organic carbonates

The organic-rich, laminated microbial boundstone (F1c; **Fig. 5A,B**) is abundant in the Khodjaipak Fm. (**Fig. 3**). This facies displays two main microfabrics: (i) 10 µm to 1 mm thick organic and micritic/microsparitic laminae that alternate with (ii) 100s of microns to 3 mm thick peloidal laminae (**Fig. 5C**). Macroscopically (**Fig. 5A**), the carbonate-rich beige laminae and the dark organic-rich laminae alternation are very similar to the intertidal microbial mats observed in the modern Abu Dhabi sabkha (Lokier et al., 2017). Microspar and micrite are abundant in both dark and clear laminae (**Fig. 5E**). Foraminifera (**Fig. 5D**) and sponge spicules rich laminae were probably formed through trapping

and binding processes (e.g., Bouton et al., 2016). OM has two main loci: (i) yellowish to dark aggregates are observed within peloids (**Fig. 5C**) and microspar crystals (**Fig. 5F**); similar coccoid bacteria clusters are documented in modern hypersaline lagoon microbialites (e.g., Dupraz et al., 2004); (ii) a thin dark lining occurs between spar crystals (**Fig. 5E,F**) and sometimes impregnates the wall of foraminifera and other microfossils (**Fig. 5D**). Three samples were observed under SEM and analysed with EDS (**Fig. 6**). Peloids develop within an alveolar OM network (**Fig. 6A-C**), very close to modern microbial mats exopolymeric substances (EPS; e.g., Dupraz et al., 2004; Pace et al., 2018). Platy clay minerals characterized by high content in Al, and possibly of detrital origin, are associated with OM (**Fig. 6D**). The clay-OM complexes include very small sulfur globules (mostly <1µm in diameter). Some samples show a significant proportion of iron oxides associated with organic carbon (**Fig. 6E,F**).

### 4.3. Bulk and clay mineralogy

Carbonates predominate in the lower and upper carbonate intervals (90-100% of CaCO<sub>3</sub>; **Fig. 3**). In the Khodjaipak Fm., the proportion of calcium carbonate and quartz fluctuates: microbial laminites (F1c) contain up to 92% of CaCO<sub>3</sub>, while brown marls to siltstones contain up to 50% of quartz. The proportion of clay minerals in the bulk rock is overall low (< 1%) in the lower and upper carbonate intervals. The percentage increases in the Khodjaipak Fm., where clay accounts for up to 8% of the bulk rock. Pyrite content is relatively low (< 0.5%) in the lower and upper carbonate intervals and reaches 1% for several horizons within the Khodjaipak Fm. (**Fig. 3**).

We subdivide the section into five intervals based on the clay mineral association (intervals 1 to 5; **Fig. 3**). From 1 m to 20 m, the relative percentage of the various clay minerals is relatively constant: illite predominates with values comprised between 55 and 75%, followed by R1 type illite/smectite mixed-layers (I/S-R1; 20-40%), kaolinite (0-10%) and chlorite (0-5%). A second interval (20 to 30 m) is characterized by an increase of kaolinite (up to 40% at 27.5 m) followed by a slight decrease, at the expense of illite (approximately 40 to 70%). Chlorite displays similar variations as kaolinite, but represents a smaller proportion of the clay assemblage (< 15%). I/S-R1 content ranges between 15 and 25%. The third interval (30 to 36 m), starting at the basis of the Khodjaipak Fm., is marked by highest content and significant fluctuations of kaolinite, with two peaks at 31 and 31.7 m, reaching 65% and

63% respectively, and the decrease of illite and chlorite contents. From 36 m to 40 m (interval 4), kaolinite and chlorite are absent, IS-R1 varies between 0 and 30%, while illite remains the dominant clay mineral.

#### 4.4. Rock Eval pyrolysis

TOC values range from 0wt% (peloidal pack- to  $\mu$ grainstone, F3a) to 6wt% for the thinly laminated black limestones with nodules (F1b; **Figs 3 and S1**). The lower carbonate interval does not contain organic carbon. Within the Khodjaipak Fm., the higher TOC values were measured in limestones (2.0% on average in F1b, F1c and F1d) while lower TOC values (avg. 0.5%) characterized marls and claystone (F1a; **Fig. S1**). The TOC is relatively low (0-1.8%) from 30 to 33.5 m, then increases from 33.5 to 34.5 m and remains at fluctuating but relatively high values (0.7-6%) up to 38 m to finally progressively decrease to 0% in the upper carbonate interval.

Throughout the dark organic-rich deposits interval, the  $T_{max}$  values form two groups (**Fig. 7**). Most of the values range between 440 and 465°C, characterizing a mature OM which has reached the oil window, while a second group shows values comprised between 515 and 540°C (**Fig. 7**), indicating the gas window (Tyson, 1995). The HI values obtained in the Khodjaipak Fm. range from 7 to 142 mgHC/g-TOC. Along the studied section, the HI fluctuates rapidly between values above 100 mgHC/g-TOC and below 50 mgHC/g-TOC (**Fig. 7**). These observations as well as the high values of  $T_{max}$  may be the reflection of a degradation of the OM. The HI /  $T_{max}$  relation (**Fig. 7**) points to a Type III (terrestrial OM) to Type IV OM, the latter characterizing a highly altered organic material (generally oxidized) whose biological origin is not determinable (Baudin et al., 2007). However the burial of OM selectively removes hydrogen-rich OM and induces a depletion of IH values (Espitalié et al., 1985). Consequently, it is probable that at least a part of the OM was originally of Type II (marine OM) and was subsequently thermally degraded. Therefore, the Rock-Eval analysis is most probably not conclusive regarding the OM origin, and palynofacies data are used to discriminate OM sources (see section 4.5).

#### 4.5. Palynofacies

The studied palynofacies comprise nine classes of components (**Fig. 8**), following a simplified version of the palynofacies classifications published by Whitaker et al. (1992), Tyson (1995) and McArthur et al. (2016): (i) opaque phytoclasts, (ii) translucent phytoclasts, (iii) dark structureless organic matter (DSOM), (iv) well-preserved wood fragments (WPWF), (v) amorphised degraded brown wood (DBW) and dark amorphous organic matter (AOM) flakes with rectangular shapes, (vi) pollen grains and spores, (vii) orange to dark AOM flakes, (viii), dinoflagellate cysts, and (ix) foraminiferal linings. The translucent phytoclasts correspond to flat, pale brown to transparent fragments including leaf cuticles and aqueous plant remains. DSOM are dark brown to orange fragments originating from degraded products of plant debris and humic gel. WPWF are dark-brown fragments showing preserved wood structures (tracheids, lignified walls). DBW and rectangular AOM are dark-brown to yellow, flat, with gelified to amorphized structures. These particles partly derive from terrestrial wood fragments and/or from lignified parts of aqueous plants that are heavily degraded, gelified and amorphized in an aqueous environment. Similar amorphization and gelification processes due to bio-degradation of terrestrial or aquatic plant tissues have been reported in peat and aquatic marshes environments (Laggoun-Défarge et al., 1999; Sebag et al., 2006; Storme et al. 2012). The vertical evolution of the relative percentage of these classes is represented on **Figure 8**.

AOM is largely predominant in the Tubiegatan section, accounting for more than 75% in the countings in 18 out of 27 samples (**Fig. 8**). AOM is generally interpreted as being of microbial and/or algal origin (e.g., Tyson, 1995) and a mostly marine origin of the AOM is probable in the Tubiegatan section. However, dark AOM particles with rectangular shape observed at some stratigraphic levels between 34.8 and 38.8 m are associated with DBW. Therefore, this type of AOM **could be** different from the rest of the observed AOM, and is here interpreted as most probably originating from degraded terrestrial and/or aquatic macrophytes tissues. A second characteristic of the palynofacies from Tubiegatan is the common occurrence of -at least partly- terrestrial-derived materials, such as opaque and translucent phytoclasts, DSOM, WPWF, amorphised DBW, and pollen and spores. This could be related to the proximity of emerged lands or islands and the relative shallowness of the sea (Carneille et al., 2018).

374 The Tubiegatan section can be subdivided into four palynofacies intervals, from base to top (**Fig. 8**):

375 -Interval I (8.4 to 27 m) is characterized by elevated proportions of small size translucent phytoclast  
376 fragments, generally > 50%, low proportions of AOM and a small fraction of opaque phytoclasts. Pollen  
377 and spores, dinoflagellate cysts and foraminiferal linings occur in traces. Interval I coincides with the  
378 top of the sequence Tu1 and the sequence Tu2 (**Fig. 8**). The large proportions of translucent phytoclasts  
379 such as cuticles and the local influx of wood fragments (opaque phytoclasts) indicate the proximity of  
380 emerged lands.

381 -Interval II (27 to 33.9 m) presents high proportions of AOM (> 90%), and  
382 minor proportions of translucent phytoclasts. Amorphized DBW, opaque  
383 phytoclasts, DSOM, WPWF, pollen and spores, dinoflagellate cysts and  
384 foraminiferal linings occur in traces. This interval corresponds to the  
385 basal part of the Khodjaipak Fm., which shows low to moderate amount of  
386 preserved OM (TOC  $\leq$  2%, TOC=0.86 % on average; **Fig. 8**).

387 -Interval III (33.9 to 39.6 m) shows alternatively predominant proportions (> 85%) of AOM and  
388 amorphized DBW and rectangular AOM flakes. Minor proportions of opaque phytoclasts occur between  
389 38 and 38.8m. DSOM, WPWF, pollen and spores, dinoflagellate cysts and foraminiferal linings occur  
390 in traces. Interval III is associated with the upper part of the Khodjaipak Fm., which shows enhanced  
391 accumulation of OM (TOC up to 6%, TOC=1.8% on average; **Fig. 8**).

392 -Interval IV (39.6 to 41 m) shows a predominance of AOM, however with lower proportions than within  
393 Interval III (AOM=61-79.7%), and moderate proportions (11.4-16%) of translucent phytoclasts. Opaque  
394 phytoclasts, pollen and spores, dinoflagellate cysts and foraminiferal linings occur in traces. It should  
395 be highlighted that the palynomorphs are not well-preserved and it is somewhat difficult to identify  
396 dinoflagellate cysts properly, especially in sample 09-E16 at 40 m. Palynofacies results from this sample  
397 should thus be taken with caution. Interval IV corresponds to the Upper carbonate interval of the  
398 Tubiegatan section, which shows low accumulation of OM (TOC=0.2% on average; **Fig. 8**).



#### 4.6. Stable isotope signals and C/N records

**Figure 9** presents the vertical evolution of  $\delta^{13}\text{C}_{\text{carb}}$ ,  $\delta^{18}\text{O}_{\text{carb}}$ ,  $\delta^{13}\text{C}_{\text{org}}$  and  $\delta^{15}\text{N}_{\text{tot}}$ . In the lower carbonate interval, the  $\delta^{13}\text{C}_{\text{carb}}$  remains positive (4-4.5‰) and relatively stable up to a small negative excursion (amplitude 0.3‰) at 14 m followed by a positive excursion (amplitude 0.7‰) at 20 m. The top of the lower carbonate interval (20 to 30 m) and the base of the Khodjaipak Fm. (30 to 33 m) record a rapid and significant decrease of  $\delta^{13}\text{C}_{\text{carb}}$  (amplitude of *ca.* 4‰) to reach values close to 0. Very negative and fluctuating  $\delta^{13}\text{C}_{\text{carb}}$  values (avg. -2.3‰) characterize the remaining part of the Khodjaipak Fm. (33-40 m). An overall decrease occurs from 33 to 38 m to reach the most negative value (-10‰), which is recorded in an organic microbial boundstone (F1c; **Fig. 10**). The signal then increases up to the top of the Khodjaipak Fm. In the upper carbonate interval, the values progressively increase but remain below 1‰. Overall, black nodular mudstones and microbial boundstones have the most negative  $\delta^{13}\text{C}_{\text{carb}}$  (respectively -4‰ and -0.7‰ on average; **Fig. 10**).

The  $\delta^{18}\text{O}_{\text{carb}}$  signal records strong variations between 0 and 12 m (amplitude of *ca.* 4‰) then progressively increases up to the top of the lower carbonate interval, with a maximum value of 0.8‰. The following Khodjaipak Fm. is marked by oscillating  $\delta^{18}\text{O}_{\text{carb}}$  values (avg. -3.4‰) combined with a stepwise decrease up to 38 m (min.: -12‰; **Figs 9 and 10**). The top of the Khodjaipak Fm. and the following upper carbonate interval are characterized by a slight  $\delta^{18}\text{O}_{\text{carb}}$  increase (max: -1.4‰).

The  $\delta^{13}\text{C}_{\text{org}}$  signal (**Fig. 9**) fluctuates slightly around -25‰ for most of the lower carbonate interval, except near the top of the Tu1 sequence (9.5 to 12 m), where the values rapidly decrease (-27.4‰) and increase to reach their maximum (-23.4‰). The signal drops from 24 to 34 m, recording its lowest value (-28.5‰) near the transition between the palynofacies intervals II and III. Then the signal slightly increases and fluctuates between -27 and -28‰ up to the top of the Khodjaipak Fm.

The  $\delta^{15}\text{N}_{\text{tot}}$  values are relatively stable in the lower carbonate interval (avg. 2.7‰). The Khodjaipak Fm. shows overall lighter  $\delta^{15}\text{N}_{\text{tot}}$  (avg. 1.7‰) with significant fluctuations, between -1.1 and 4.5‰. The lowest values are recorded within the two anoxic intervals (**Fig. 9**).

The C/N ratio (**Fig. S3**) fluctuates between 5.5 and 81.9 (avg. 14.7) along the lower carbonate interval. The variations increase in amplitude (8.6 to 187; avg. 39.2) in the Khodjaipak Fm.

#### 4.7. Major and trace elements

Most of major elements (Si, Fe, K, Ti) and many trace elements (Cr, Co, Th, Pb) show strong correlation ( $R > 0.9$ ) with Al and are thus associated with the detrital fraction. Only few elements (Cd, Mo, Ni, U, V, Zn) are not ( $R < 0.5$ ) or only slightly to moderately ( $R < 0.9$ ) correlated with Al and show noticeable positive values of fraction in excess. This is particularly true for Mo, U and to a lesser extent V, which are particularly sensitive to oxygen variations. An increase of their concentrations compared with the upper continental crust values (1.5 ppm for Mo, 107 ppm for V and 2.8 ppm for U; McLennan, 2001) point to  $O_2$ -depleted conditions (Tribovillard et al., 2006; L  zin et al., 2013). **Here, only the evolution of  $Mo_{xs}$  will be shown and discussed, U and V having the same evolution (Fig. 11).** In the Khodjaipak Fm., punctual peaks of  $Mo_{xs}$  are observed, one at 34.5 m (73.2 ppm) and two others are observed at 37.2 and 38.3 m (with values up to 75.3 ppm; **Fig. 11**). In the second interval, the highest values of  $Mo_{xs}$  are related to the microbial boundstone (F1c).

To better constrain the  $O_2$  variations, U/Th and V/Cr ratios were calculated. They are both used as oxygenation tracers at the water-sediment interface or in the sediment. Based on threshold values, defined by Jones and Manning (1994), these ratios allow to distinguish oxic ( $U/Th < 0.75$  and  $V/Cr < 2.00$ ), dysoxic and anoxic ( $U/Th > 1.25$  and  $V/Cr > 4.25$ ) conditions. In the Khodjaipak Fm., the values of ratios range from 0.95 to 8.00 for U/Th and from 1.40 to 13.40 for V/Cr (**Fig. 11**), indicating  $O_2$ -depleted conditions. Two intervals are characterized by high values of these ratios attesting for anoxic conditions: the AI1 (for Anoxic Interval n  1) extends from 33.4 to 35.0 m, the AI2 from 37.0 to 38.7 m (**Fig. 11**). The two anoxic intervals coincide with most of the elevated TOC values. The lower values below AI1, between AI1 and AI2 and above AI2 reflect dysoxic conditions.

## 5. DISCUSSION

### 5.1. Signal preservation and signification

#### 5.1.1. Organic matter

The  $T_{\max}$  from Rock-Eval analyses indicate the influence of a burial diagenesis consistent with the oil window (**Fig. 7**). The HI and OI values plotted in the Van Krevelen diagram (**Fig. S2**) suggest the presence of altered OM (Type IV) and/or continental (Type III) OM. Nevertheless, in the case of thermal diagenesis associated with marked burial of the sediments, the hydrogen bonds of organic molecules are particularly affected, leading to a decrease of the original HI values (Espitalié et al., 1985). HI values were thus originally higher than recorded, probably mostly fitting with a Type II (marine) OM, as reflected by predominant AOM in palynofacies. Furthermore, the local presence of orange-colored AOM, combined with a relatively low amount of pyrite (< 2%) could reflect an early diagenetic sulfurization of OM (Tribovillard et al., 2004). This process is widespread in anoxic marine environment and implies reactions between dissolved sulfide species and energy-rich moieties in the OM (e.g., alcohols, aldehydes). Sulfurized polymers (mostly lipids) are more resistant to microbial and/or thermal degradation (Raven et al., 2019), which could explain the preservation of substantial amounts of OM in the Khodjaipak Fm. The low OI values (**Fig. S2**) also suggest that most of the OM of the Khodjaipak Fm. is a thermally degraded Type II OM (Espitalié et al., 1985; Tyson, 1995). Accordingly, the measured TOC values are lower than the original ones. As TOC values reach up to ~6% in the Khodjaipak Fm., this implies high to very high original OM accumulation for a marine depositional environment. Despite a significant burial, the consistency of palynofacies, organic carbon geochemistry and sedimentary facies indicates that the OM retains part of the paleoenvironmental signal.

#### 5.1.2. Clay minerals

The presence of kaolinite is compatible with limited burial temperatures, probably less than 160°C (Sucha et al., 1993). Dellisanti et al. (2010) estimated that for  $T_{\max}$  ranging from 434 to 465°C, the content of illite in the IS-R1 should vary between 60 and 85%. In this context the illitization of potential former smectites or smectite-rich interstratified clay mineral such as IS-R0 may have occurred, these transformations taking place around 80-100°C, i.e. from 2 to 3 km of burial for a normal geothermal

gradient (Chamley, 1989). Both clay mineral assemblages and the maturity of the OM agree therefore with a minimal burial depth of *ca.* 2-3 km for the Khodjaipak Fm., if we consider a normal geothermal gradient. Occurrence of initial smectite-rich clay minerals in the initial sediment is therefore a possibility and could correspond to current IS-R1. Preservation of kaolinite less sensitive to burial diagenesis allows to use these detrital clay minerals for palaeoclimate reconstruction, as no evidence of clay mineral authigenesis was recognized.

### 5.1.3. Carbonates

The petrographic examination of the lower carbonate interval and of the Khodjaipak Fm. deposits points to moderate chemical and/or mineralogical diagenetic transformations. The predominantly micritic facies of the lower carbonate interval show very small quantities of carbonate cements, except for late fracturation (e.g., **Fig. 4B**). The Khodjaipak Fm. is also cross-cut by late fractures infilled with fibrous gypsum, possibly formed through precipitation of sulfate-rich fluids coming from the overlying sabkha gypsum (**Irgayli Fm.; Fig. 3**). Microbial facies are predominantly micritic to microsparitic (**Fig. 6**). This probably indicates a moderate cementation and recrystallization of the carbonate minerals. The negative  $\delta^{18}\text{O}_{\text{carb}}$  values comprised between 1‰ to -5‰ with two values reaching -8‰ and -12‰ (**Figs 9, 10**) indicate a potential contribution of meteoric water. However, in modern microbialites, the micrite to microspar transition can take place in the depositional environment. It is not necessarily related to meteoric cementation, but can also result from physico-chemical precipitation in alkaline water close to the microbialite surface, though the resulting isotopic values are generally not very negative (Dupraz et al., 2004, 2013). By contrast, the upper carbonate interval shows an intense cementation and a poor preservation of the facies, which indicates a probable strong diagenetic influence.

The effects of recrystallization by fluids circulation could affect the isotopic signal, particularly the  $\delta^{18}\text{O}_{\text{carb}}$  primary signal, which is more sensitive than the  $\delta^{13}\text{C}_{\text{carb}}$  (Immenhauser et al., 2002; Louis-Schmid et al., 2007). A strong covariation of  $\delta^{13}\text{C}_{\text{carb}}$  and  $\delta^{18}\text{O}_{\text{carb}}$  usually indicates the influence of meteoric fluids (e.g., Marshall, 1992). **Figure 10** presents a  $\delta^{13}\text{C}_{\text{carb}}$  vs.  $\delta^{18}\text{O}_{\text{carb}}$  cross plot. The linear least square regressions were calculated for the lower carbonate interval ( $R^2 = 0.3$ , P-value < 0.05) and

the Khodjaipak Fm. ( $R^2 = 0.3$ , P-value < 0.05). The results confirm a possible significant diagenetic imprint on the  $\delta^{18}\text{O}_{\text{carb}}$ , which is consequently not used for palaeoclimate reconstructions.

#### 5.1.4. Sulfides and iron oxides

Given their morphology and their association with OM-clay complexes, the very small sulfur-rich globules (< 1  $\mu\text{m}$  in diameter) that are observed in microbial boundstones (**Fig. 6D**) are likely of microbial origin. They are very similar to the globules produced by sulfide-oxidizing anoxygenic phototrophic bacteria in modern hypersaline microbial mats (e.g., Pace et al., 2018). The Khodjaipak Fm. also shows moderate amounts of pyrite (< 1.5% of the bulk sediment) decreasing upward (**Fig. 3**). Pyrite is a common byproduct of microbial sulfate reduction and is common in organic-rich sedimentary formations (e.g., Rickard et al., 2017; Raven et al., 2019). The relatively low proportions of pyrite characterized here could be the result of the competition between reactive sulfides and OM for reactive Fe shortly after deposition (Shawar et al., 2018). A significant OM production, as postulated for the Khodjaipak Fm., could constitute a Fe sink and prevent pyrite precipitation (see section 5.3.3). At the micro-scale, the Fe oxides locally associated with OM (**Fig. 6F**) could derive from the oxidation of pyrite originally associated to OM-clay complexes. Fibrous gypsum filling fractures testifies of the post-depositional circulation of sulfate-rich fluids in the rock. These oxidizing fluids, probably coming from the overlying gypsum deposits (Irgayli Fm.; **Fig. 2**), could have induced the oxidation of primary pyrite. Overall, the top of the Khodjaipak Fm. is more affected by the circulation of oxidizing, probably saline fluids than its base.

#### 5.2. Age of the Khodjaipak Fm. and stratigraphic relationship with the SW Gissar carbonate platform

Based on ammonite biostratigraphy, Abdullaev (2004) dated the Khodjaipak Fm. as Late Oxfordian to Kimmeridgian, while Mitta and Besnosov (2007) suggested a Late Oxfordian to Early Kimmeridgian age. The ammonite fauna identified in this study indicates a Middle Oxfordian age at least in Tubiegatan (**Fig. 3**). *Dichotomosphinctes luciaeformis* indicates an older age for the top of the lower carbonate interval (Transversarium Zone, Luciaeformis subzone), while *Subdiscosphinctes* (*Subdiscosphinctes*) cf.

*richei* De Riaz /*mindowe* Siemiradzki gives a more recent age (Transversarium zone, Schilli subzone) in the upper part of the Khodjaipak Fm. The fact that all these deposits belong to two successive subzones also indicates that there is no major stratigraphic hiatus associated with the hardground at the top of the lower carbonate interval (Fig. 3).

A vast carbonate platform developed over the SW Gissar between the Early Callovian and potentially the Kimmeridgian (Abdullaev, 2004; Nugmanov, 2010; Carmeille et al., 2018). Carmeille et al. (2018) subdivided the platform succession in two major sequences: (i) a ramp developed during the Callovian Sequence (CS; equivalent to the Lower Kugitang Fm. of Nugmanov, 2010), which is dated as Early-Middle Callovian with ammonites, and possibly includes Upper Callovian deposits; (ii) The Upper Jurassic Sequence (UJS; equivalent to the Upper Kugitang Fm. of Nugmanov, 2010) develops above a major sequence boundary (SB2) and after a hiatus potentially encompassing the top of the Callovian and the base of the Oxfordian (Fig. 2). In the SW Gissar, the UJS is dominated by mud or oncoid-rich lagoonal deposits (the Gardarin Fm. of Abdullaev et al., 2010) passing southward to ooid shoal grainstones and peri-reefal facies. Carmeille et al. (2018) proposed a correlation between several  $\delta^{13}\text{C}_{\text{carb}}$  excursions recorded in the UJS and the well-dated NW Tethyan bivalve  $\delta^{13}\text{C}$  record compiled by Dera et al. (2011). This correlation would date the UJS as Early to Late Oxfordian. The stratigraphic relationships between the SW Gissar carbonate platform and the Khodjaipak Fm. are key to understand the palaeogeographic setting and to establish a depositional model for the organic carbonates.

Some authors (e.g., Abdullaev, 2004; Nugmanov, 2010) propose a model where organic shales (Khodjaipak Fm.) accumulate in a starved basin separated from the lagoon (Gardarin Fm.) by a large-scale barrier reef (Urtabulak Fm.). Besides the presence of Oxfordian ammonite specimens recognized in the Khodjaipak Fm., the lower carbonate interval of the Tubiegatan section shows heavy  $\delta^{13}\text{C}_{\text{carb}}$  values (4.25‰ on average), which largely overlap with the values measured in the UJS lagoonal deposits (4.00‰ on average; Fig. 10). Consequently, the lower carbonate interval, underlying the Khodjaipak Fm. could be time equivalent of part of the UJS inner shelf deposits in the SW Gissar.

The UJS is subdivided into two medium-scale sequences, UJS1 and UJS2, which are separated by a major sequence boundary which resulted in the exposure of the northern part of the SW Gissar platform

(SB3; **Fig. 12**; Carmeille et al., 2018). We propose two correlation hypotheses between the organic-rich Tubiegatan succession and the SW Gissar inner platform deposits, based on the carbon-isotope signal and the sequence stratigraphy.

### 5.2.1. Hypothesis n°1

The first hypothesis (**Fig. 12A**) correlates the  $\delta^{13}\text{C}_{\text{carb}}$  positive peak (*ca.* 0.5-1.2‰) recorded in the middle part of UJS1 in the Baglydara and Pyandzh Ob sections with a similar excursion in the lower carbonate interval of the Tubiegatan section (*ca.* 0.5‰ of amplitude, **Fig. 12A**). Above, a gradual decrease of  $\delta^{13}\text{C}_{\text{carb}}$  is recorded up to SB3, leading to a marked negative excursion in the  $\delta^{13}\text{C}_{\text{carb}}$  (amplitude > 0.5‰ in Baglydara, > 2‰ in Pyandzh Ob and *ca.* 1‰ in Derbent; **Fig. 12A**). The  $\delta^{13}\text{C}_{\text{carb}}$  then progressively increases of 1.5 to 2‰ above SB3. In Tubiegatan, the  $\delta^{13}\text{C}_{\text{carb}}$  shows a decrease of *ca.* 4‰ encompassing the top of the lower carbonate interval and the basis of the Khodjaipak Fm. Then, the values drop down to -10‰ in Khodjaipak Fm. before a progressive increase (amplitude > 10‰) from the top of the Khodjaipak Fm. to the upper carbonate interval. Hypothesis n°1 implies a correlation of these two negative peaks of different amplitude. In this scenario, the Khodjaipak Fm. would have deposited at a period of low sedimentation rate (recorded by burrowed horizons) and partial subaerial exposure of the platform associated with SB3 (Carmeille et al., 2018).

### 5.2.2. Hypothesis n°2

In the second hypothesis, we correlate the slight positive excursion recorded in Tubiegatan with three similar excursions (amplitude of *ca.* 1‰) recorded in the first half of UJS2 in the SW Gissar sections (blue colour, **Fig. 12B**). This is followed by rapid 1 to 1.5‰ fluctuations in the  $\delta^{13}\text{C}_{\text{carb}}$  in Baglydara or by 1 to 2‰ negative carbon-isotope excursions in Pyandzh Ob and Derbent, about 40–60 m under the nodular gypsum strata of the SW Gissar platform (**Fig. 12B**). The same negative excursion was recorded below evaporites in other SW Gissar sections (e.g., Langar; Carmeille et al., 2018). In Derbent, the top of the carbonate succession shows a *ca.* 2‰ positive excursion in the  $\delta^{13}\text{C}_{\text{carb}}$  record. This excursion can be correlated with the positive excursion recorded at the top of the Khodjaipak section. Hypothesis n°2 is supported by sedimentological arguments. UJS2 displays several bioturbated horizons (e.g., in Baglydara and Derbent sections). Similar burrowed limestones are also observed along the lower



carbonate interval in Tubiegatan, at Tu1 and Tu2 sequence boundaries (**Figs 3, 12**). In the proposed correlation, the burrowed horizons would be approximately in the same stratigraphic interval as in Tubiegatan. Inner platform stromatolites and pellet-rich deposits form the transition between the organic carbonates (Khodjaipak Fm.) and the overlying nodular gypsum. Similarly, UJS2 is overlain by sabkha gypsum over the whole SW Gissar (**Fig. 12**), which reflects a progressive restriction and an increasing salinity on the platform (Carmeille et al., 2018). In this scenario, the organic carbonates would form contemporaneously of an evaporative, low energy carbonate lagoon rich in gypsum pseudomorphs (**Fig. 12**). This second hypothesis appears to be the most likely scenario, and will be used throughout the paper.

### **5.3. Sedimentological and geomicrobiological models for the Khodjaipak organic carbonates**

#### **5.3.1. Depositional environment of the organic carbonates**

The fine-grained textures, conjugated with abundant and thin microbial laminations (**Figs 3 and 5**), suggest a low-energy depositional environment for the Khodjaipak organic sediments (Droste, 1990; Warren, 2011). The intercalation of thin shell beds (**Figs 3 and 4F**) indicates the punctual influence of hydrodynamic events, probably storms, in this predominantly quiet environment. The presence of early diagenetic sulfate pseudomorphs (**Fig. 5B**) may also indicate punctual hypersaline conditions within the sediment (Warren, 2011). The Khodjaipak Fm. organic carbonates are overlain by **intertidal to shallow subtidal** lagoon carbonates and then by supratidal sabkha chicken-wire gypsum (**Figs 1C and 3**). Similar successions have been documented in the shallow subtidal to supratidal sequences of the Holocene Abu Dhabi sabkha (e.g., Lokier and Steuber, 2008; Aref and Taj, 2018), but also in the Upper Jurassic to Lowest Cretaceous Purbeck beds (northern France-southern UK; e.g., Shearman, 1966; West, 1975; Schnyder et al., 2009) or in the Upper Permian of the Dolomites (Italy; e.g., Hardie, 1986). Jahnert and Collins (2012) and Bouton et al. (2016) have also shown that thin storm shell beds can be trapped and preserved within shallow water (< 1 m deep) hypersaline lagoon microbialites from Australia (Shark Bay) and Cuba (Cayo Coco). In the absence of subaerial exposure evidence, we postulate that the Khodjaipak Fm. was deposited in a shallow subtidal environment (**Fig. 13A**). This would imply that



part of the pelagic fauna (e.g., ammonites; **Figs 4 and 5**) could have undergone a post-mortem drift (e.g., Olivero and Raffi, 2018). The documented benthic foraminifera (e.g., *Lenticulina* and *Reinholdella*) are opportunistic, well adapted to low oxygen levels and can live in shallow water (Rita et al., 2016). A shallow environment (depth of *ca.* 30 m) was hypothesized for the deposition of the organic-rich part of the Middle-Upper Oxfordian Hanifa Fm. on the Arabian Platform (Warren, 2011). Relatively shallow seas and the proximity of emerged lands or islands during the Khodjaipak Fm. deposition are also consistent with the common occurrence of terrestrial-derived materials in palynofacies, such as opaque and translucent phytoclasts, DSOM, WPWF, amorphized DBW, and pollen and spores.

The highest TOC values are found in nodule-rich carbonate facies (F1b; **Fig. S1**). On the opposite, quartz-rich siltstones have relatively low TOC, which indicates that carbonate production is associated with OM concentration, while detrital influx probably favored OM dilution. Palynofacies yielding opaque and translucent phytoclasts between 38 and 41 m (**Fig. 8**) indicate an increase of terrestrial organic inputs at the top of the Khodjaipak Fm. (**Fig. 13A**). This interval rich in terrestrial phytoclasts occurs after the decline in kaolinite proportion (**Fig. 3**) and precedes sabkha gypsum deposition. During the Middle-Late Jurassic, periods marked by drastic changes in kaolinite are often interpreted as variations in humidity conditions: high proportions in kaolinite point to humid conditions and low kaolinite proportions to a drier climate (Pellenard and Deconinck, 2006; Schnyder et al., 2006; Brigaud et al., 2014; Pellenard et al., 2014). Part of the opaque phytoclasts could also derive from wildfires in the nearby continental areas. All these observations indicate that a climate aridification occurred during the deposition of the Khodjaipak Fm. and could have culminated with the platform-wide development of sabkha (Irgayli Fm.; **Figs 2, 12 and 13A**; Carmeille et al., 2018).

### 5.3.2. Geomicrobiological model

The thinly laminated microfabrics (**Fig. 5**), the presence of peloids and intra-carbonate OM (**Figs 5C and 5E**), the potential preservation of EPS remnants (**Fig. 6C**) and the abundance of AOM in palynofacies (**Fig. 8**), all suggest that most of the OM was produced by microorganisms (bacteria, archaea and/or unicellular algae), notably in benthic microbial mats. The preservation of microbial

lamination can be partly explained by the scarcity of bioturbation, which in turns reflects the low  
 oxygenation of the bottom sediment (Warren, 2011). Trace elements concentrations and ratios indicate  
 alternating anoxic and suboxic conditions (**Fig. 11**). The potential development of contemporaneous  
 organic-poor carbonates in the SW Gissar (**Figs. 12 and 13A**) could indicate water stratification, where  
 the Khodjaipak Fm. accumulated in poorly oxygenated bottom water. These O<sub>2</sub>-depleted conditions  
 promoted the preservation of OM -possibly through early sulfurization- and supported the development  
 of anaerobic microorganisms in the mats (e.g., methanogenic and methanotrophic microbes; **Fig. 13B**).  
 The two anoxic episodes are associated with the highest TOC, but also with the lowest  $\delta^{13}\text{C}_{\text{carb}}$  and  
 $\delta^{15}\text{N}_{\text{tot}}$  (**Fig. 9**). During these episodes, we suggest that extensive denitrification decreased nitrate  
 availability and therefore, phytoplankton (including cyanobacteria) capable of using an alternate source  
 of nitrogen such as N<sub>2</sub> may have become quantitatively important, leading to a  $\delta^{15}\text{N}_{\text{tot}}$  values close to  
 zero (**Fig. 9**; Quan and Falkowski, 2009). In addition, the dinitrogen gas increase could be a consequence  
 of the anammox bacterial activity, which converts ammonium from the anoxic water to N<sub>2</sub> by anaerobic  
 metabolic reaction (**Fig. 13B**; Kuypers et al., 2003; Kuenen, 2008).

In shallow hypersaline lagoons, the majority of OM derives from photosynthesis (Warren, 2011).  
 However, there is only indirect evidence for the presence of photoautotrophic microorganisms in the  
 Khodjaipak Fm. Sulfur-rich globules in OM-clay complexes (**Fig. 6D**) could indicate the activity of  
 anoxygenic phototrophs such as purple sulfur bacteria (Pace et al., 2018). Whether oxygenic  
 photosynthesis occurred in the Khodjaipak Fm. remains uncertain. The micritic and microsparitic  
 peloids observed in microbial boundstones (F1c; **Figs 5C and 6A to 6D**) are typically related to  
 microbial degradation of EPS. In modern mats, the degradation of some EPS components by  
 heterotrophic bacteria, e.g., sulfate-reducing bacteria (SRB), leads to the formation of carbonate peloids  
 by increasing alkalinity and releasing cations adsorbed by EPS such as calcium (e.g., Dupraz et al.,  
 2004; Visscher and Stolz, 2005; Pace et al., 2018). The presence of sulfate pseudomorphs in the  
 Khodjaipak Fm. (**Fig. 5B**) indicates that porewaters were sulfate-rich. Sulfates could have been reduced  
 by SRB to (hydrogen) sulfides (**Fig. 13B**) and part of the sulfides could have been subsequently  
 precipitated as pyrite or bound to part of the EPS through OM sulfurization. The study conducted by

Arp et al. (2008) in the Upper Jurassic of north Germany documents calcitized pseudomorphs of gypsum with **very** low  $\delta^{13}\text{C}_{\text{carb}}$  within stromatolites, suggesting oxygen-depleted sulfate-bearing environments associated with high rates of methane production. These authors made the hypothesis that the gypsum was replaced by magnesian calcite at the transition of suboxic and sulfidic zones within the mats **during sulfate-driven anaerobic oxidation of methane (SD-AOM; Reeburgh, 2007; Himmler et al., 2018)**. The  $\delta^{13}\text{C}_{\text{carb}}$  values measured in the Khodjaipak Fm. (ranging from -10‰ to +2‰) are not negative enough to support this process, but fit well with the  $\delta^{13}\text{C}_{\text{carb}}$  range of hypersaline lake microbial mats with a high organic productivity and SRB activity (Vasconcelos et al., 2006; Glunk et al., 2011; Dupraz et al., 2013). Both geochemical and petrographic data suggest a strong contribution of microbial sulfate reduction to carbonate precipitation. However, SD-AOM cannot be totally ruled out. Indeed, a late recrystallization of carbonates in e.g., meteoric water, or a contribution of bioclast dissolution could have led to an increase of the original  $\delta^{13}\text{C}_{\text{carb}}$  values (e.g., De Boever et al., 2009).

### **5.3.3. Regional depositional model**

On epeiric platforms, OM accumulates in two types of mesohaline settings, often in association with evaporites (Warren, 2011): (i) intrashelf basins or (ii) shelf edge depressions. Both cases imply a tectonically and/or climatically-driven restriction of shelf water and the formation of a hydrographic barrier partially isolating a **water body, leading to the formation of stratified brines**. In the Oxfordian Hanifa Fm., reef or bioclastic platform rims built during transgressions, and subsequently isolated the intrashelf basins during sea level falls (Carrigan et al., 1995; Warren, 2011). The Late Jurassic semi-arid climate **also enhanced the** formation of anoxic/dysoxic saline brines in coastal lagoons and lakes from the Purbeck Beds of Dorset (UK, Late Tithonian-Berriasian). Combined with freshwater inputs linked to a climatic change toward slightly more humid conditions during the Berriasian, this ultimately led to the development of water stratification and the deposition of OM-rich facies linked to enhanced primary productivity in surface waters and its preservation in anoxic bottom water brines (Schnyder et al., 2009). Similar examples of coastal marine sediment enriched in OM are known from Holocene **coastal salinas and** hypersaline lagoons of Australia (Bauld, 1981; McKirdy et al., 1992; Coshell and Rosen, 1994).

The Kugitang cliffs, located *ca.* 10 km south of Baglydara and 35 km east of Tubiegatan, display the largest reefs outcropping in the SW Gissar (up to 80 m high; Ilyn and Fortunatova, 1988). These reefs could be a stratigraphic equivalent of UJS1, where peri-reefal bioclastic deposits were documented (**Fig. 13A**; Carmeille et al., 2018). Such reefs complexes could have formed a hydrographic barrier limiting water exchanges between the oxygenated inner platform water and the stratified brines in Tubiegatan. Based on the correlation hypotheses **proposed Figure 12**, we built a four steps **carbonate shelf to anoxic intrashelf lagoon** evolution scenario which shares some similarities -but is not strictly equivalent- with that of the Purbeck Beds of Dorset or that of the above-mentioned Holocene Australian coastal lakes and lagoons (**Fig. 13A**):

**-step 1:** the facies of the lower carbonate interval (sequences Tu1, Tu2 and the basis of Tu3) show evidence for a good oxygenation of the water column. At that time, it is unknown whether Tubiegatan was located on a seaward opened mid to outer shelf or within a well oxygenated, non-stratified lagoon. At the top of the lower carbonate interval (Tu3 transgressive cycle, *Luciaeformis* SZ, *Transversarium* Zone, Middle Oxfordian, **Fig. 3**), a flooding associated with a low sedimentation rate could explain the formation of a subaqueous hardground. Reworking and erosion of coastal vegetalized areas due to the transgression led to local inputs of wood debris, as indicated by opaque phytoclasts occurrences in the palynofacies (**Fig. 8**). An intrashelf lagoon could have formed following the development of two hydrographic barriers: (i) eastward, large coral reefs located to the south of the SW Gissar; (ii) a hypothetical second hydrographic barrier located westward and/or southward (not outcropping).

**-step 2:** the presence of detrital kaolinite, usually formed during pedogenesis in intertropical climate, suggests enhanced **water** precipitation at the beginning of the Khodjaipak Fm. deposition (*Luciaeformis* to *Schilli* SZ, *Transversarium* Zone, Middle Oxfordian). Freshwater inputs, combined with limited lagoon to basin water exchange leads to progressive water stratification. Extensive microbial mats developed in the suboxic brines at the bottom of the intrashelf basin. Developing **benthic and possibly planktonic** algal/bacterial productivity led to a relative increase in OM deposition ( $\text{TOC} \leq 2\%$ ), with palynofacies dominated by AOM. Shell beds may have derived from the reworking of peri-reefal sediments by storm currents leading to possible punctual re-oxygenation of the bottom water mass.

-step 3: a regional aridification is attested by the disappearance of kaolinite and increase of IS-R1 possibly through illitization of original smectite, and characteristic of semi-arid climate and possible wildfires as indicated by opaque wood fragments input. **The associated sea level drop** led to the exposure of the reef complex and to an increasing isolation of the intrashelf basin, which became near permanently anoxic (**Fig. 13A**; Schilli Subzone, Transversarium Zone, Middle Oxfordian). Step 3 is also the period of maximum microbial mat development. The climatic conditions might still allow some periodic freshwater inputs supporting primary productivity in surface waters and the local development of ligneous aquatic or emerged coastal plants producing AOM and DBW, respectively (**Fig. 8**). The shallow, restricted water may have promoted in situ amorphization/gelification processes of the DBW. As a result of enhanced organic productivity (possibly from organic various sources) and developing stratification, anoxia and OM deposition reached a maximum (TOC up to 6%).

-step 4: the increasing evaporation progressively decreasing water depth and lowering freshwater input, could have limited water stratification. The resulting mixing of surface and bottom water led to the oxygenation of the bottom of the intrashelf basin **and to OM oxidation** ( $\text{TOC} \leq 1\%$ ; **Fig. 8**). Intertidal to shallow subtidal lagoon deposits, **including stromatolites**, prograded and progressively filled the accommodation space (Schilli to post-Schilli Subzones, Middle Oxfordian). **Finally the nodular gypsum accumulation records the development** of a large scale sabkha covering the whole SW Gissar region (**Fig. 3**). These sabkha deposits are either anterior to- or partly contemporaneous with the thick salt-anhydrite series of the Gaurdak Fm. (Kirshin, 2007; Abdullaev et al., 2010; Carmeille et al., 2018). At this time, regional subsidence is very low (Brunet et al., 2017). Compression and uplifts in the Afghan and Central Iranian blocks related to the closure of the Waras-Panjaw Ocean (Central Afghanistan) probably leads to the restriction of the ADB and to the deposition of the Gaurdak salt (Barrier et al., 2018). Overall, the organic-rich deposits of Khodjaipak Fm. may record the onset of the isolation of the ADB from more open areas (e.g., the South Caspian Basin). In this case the new datings provided by ammonites identification at Tubiegatan would demonstrate that this disconnection would have begun at the end of the Middle Oxfordian. This tectonic disconnection, combined with a regional aridification

could have led to the progressive increase of evaporation and subsequent organic-rich deposits in intrashelf depressions on the carbonate platform.

#### **5.4. Context and origin of worldwide-scale organic-rich accumulations during the Oxfordian-Kimmeridgian**

The Early Oxfordian-Early Kimmeridgian period is characterized by the multiplication of organic-rich deposits worldwide and could be considered as a Late Jurassic Ocean Anoxic Event as proposed by Trabucho-Alexandre et al. (2012) and Martinez & Dera (2015). Major source rocks developed during this period, e.g., the Hanifa Fm., the Kimmeridge Clay Fm., the Smackover Fm. (Warren, 2011). **Figure 14** compiles the facies and composition of these organic-rich formations. On the Arabian Platform, the Lower to Upper Oxfordian organic-rich Hanifa Fm. accumulated in small and semi-isolated intrashelf basins, where seawater was stratified and OM accumulated in dysoxic to anoxic bottom water (Droste, 1990; Carrigan et al., 1995; Vahrenkamp et al., 2015). The progressive isolation of these basins could be the consequence of an uplift of the Eastern margin of the Arabian Plate. The restriction of the intrashelf basins subsequently led to the evolution from normal marine shallow-water carbonates during the Kimmeridgian (Jubaila Fm.) to restricted-marine and evaporitic deposits during the Tithonian (Arab A, B, C and Hith Fms). In Wadi Nisah (Saudi Arabia), the Hanifa Fm. is composed of fine-grained carbonates and biosiliceous facies (Hawtah Member) overlain by organic-rich silty carbonates alternating with burrowed skeletal facies (Ulayyah Member; Eltom et al., 2017). These deposits are interpreted to reflect the circulation of nutrient-rich currents derived from shelf edge upwellings into the intrashelf basins (Eltom et al., 2017). The basal sequence C of the Smackover Fm. and its distal equivalent, the Haynesville Shales, are major source rocks in the United States Gulf Coast. These formations include organic-rich (up to 6% TOC) and biosiliceous lime mudstones, which accumulated in anoxic intrashelf basins delimited by carbonate platforms and islands (Heydari et al., 1997; Hammes et al., 2011; **Fig. 14**). Some of these facies could be of microbial origin (Hammes et al., 2011). In the Neuquen Basin (Argentina), the unit 1 of La Manga Fm. consists of basinal organic rich shale-limestone **sequences** (up to 4.5% TOC), which are locally rich in radiolarians (Palma et al., 2014). These deposits could have accumulated in half grabens, close to the hanging wall of normal faults, while shallow water

carbonates could have been produced on the foot wall. They are overlain by Middle-Upper Oxfordian intertidal stromatolites with fenestrae and mud-cracks (Palma et al., 2015; **Fig. 14**) and finally by sabkha deposits of the Auquilco Fm., recording an overall shallowing trend. A common stratigraphic pattern characterizes these Middle to Upper Oxfordian organic-rich formations: (i) subtidal normal marine carbonates are overlain by (ii) mesohaline organic facies (often microbial). This succession reflects the progressive stratification of the water, sometimes associated with- or followed by an increase of the salinity. Then, (iii) intertidal to shallow subtidal carbonates are overlain by (iv) sabkha deposits, indicating the filling of the accommodation space and a further increase of salinity. The quasi-contemporaneous formation of intrashelf basins on the northern and southern margins of the Tethys, in the Gulf of Mexico and on the eastern margin of the Pacific during the Middle to Late Oxfordian could reflect the early stages of the large-scale compressive tectonic events known as Late Cimmerian or Neo Cimmerian in western central Asia (Late Jurassic to Early Cretaceous; i.e. Boulin, 1990) leading to margin inversions.

The influence of these multiple organic accumulations on the global carbon cycle can be questioned. In the subalpine basin, a short-term negative  $\delta^{13}\text{C}_{\text{carb}}$  excursion (*ca.* 0.5-2‰) in the Transversarium Zone punctuates a long-term positive trend encompassing the Plicatilis–Transversarium interval (Padden et al., 2001; Louis-Schmid et al., 2007; Rais et al., 2007). A negative excursion was also measured in nodular micritic limestones known as Knollenkalk in the Swiss Jura Mountains (Rais et al., 2007; **Fig. 14**). These excursions could be the result of gas hydrate dissociation (Jenkyns, 2003). The compilation of western Tethyan belemnite  $\delta^{13}\text{C}$  by Martinez and Dera (2015) shows a major negative carbon isotope excursion (amplitude of *ca.* 4‰) at the Middle to Late Oxfordian transition (Transversarium-Bifurcatus Zone; **Fig. 14**). This period coincides with a maximum in the high eccentricity of the 9 Myr orbital cycle as defined by Martinez & Dera (2015). According to these authors the maxima of eccentricity leading to altered climatic conditions. Stratification of the water and anoxia develop preferentially during seasonal humid phases, inducing terrestrial runoff and nutrient inputs. During arid phases, increasing evaporation leads to homogenization of the water mass (Martinez and Dera, 2015). The sedimentological, geochemical and mineralogical evolution of the Khodjaipak Fm. (**Fig. 13A-B**) fits

well with this scenario. In addition, the CO<sub>2</sub> outgassing from protracted magmatic activity along spreading ridges and subduction zones could have induced warming climate during the Late Jurassic, leading to increase the flux of nutrients and, as a consequence, the biological productivity (e.g., Jones and Jenkyns, 2001; Georgiev et al., 2017). However, no lines of evidence of this long-lived volcanic activity were still published. Overall, the multiplication of OM storage events during the top of the Middle Oxfordian and the base of the Upper Oxfordian (**Fig. 14**) could have resulted from the conjunction of (i) an orbital-driven, changing seasonal climate coupled to (ii) an increase in continental and/or oceanic nutrient inputs and (iii) the initiation of tectonic inversion on several margins.

## CONCLUSION

The Khodjaipak Fm. is composed of organic-rich carbonates, marls and siltstones which were deposited on a vast part of the Amu Darya Basin. These deposits were newly dated to the Middle Oxfordian, Transversarium Zone (Luciaeformis and Schilli Subzones). The predominant facies are organic laminated siltstones, nodular mudstones reaching ~6% of total organic carbon, and thinly laminated microbial boundstones. These organic-rich deposits are overlain by shallow water lagoon and sabkha deposits. Palynofacies and petrographic analyses show that a significant proportion of the OM was produced in benthic microbial mats together with possible secondary contributions of algal/bacterial primary production in surface waters and local developments of aquatic macrophytes. Rock-Eval pyrolysis analyses indicate that the OM is mature (reaching the oil window), and that a minimal burial depth of 2-3 km was reached. The concentrations and ratios of trace metallic element record two anoxic episodes punctuated by dysoxic periods. These anoxic episodes are associated with the highest total organic carbon values, enhanced aquatic primary production (possibly from several sources as indicated by palynofacies),  $\delta^{15}\text{N}_{\text{tot}}$  close to zero and the most negative  $\delta^{13}\text{C}_{\text{carb}}$  values. Both microfabrics and palynofacies indicate a significant contribution of microbial communities to the formation of carbonates. The presence of sulfate pseudomorphs, sulfur globules and pyrite could indicate bacterial sulfur cycling. The light  $\delta^{13}\text{C}_{\text{carb}}$  signature of stromatolites and carbonate nodules could be related to precipitation influenced by sulfate reduction. Overall, the Khodjaipak Fm. is interpreted to have formed in a shallow water stratified, probably slightly hypersaline (intrashelf?) basin, periodically isolated from the



contemporaneous lagoon by pre-existing carbonate platform reliefs (e.g., coral reefs). The Middle-  
Upper Oxfordian is characterized by the multiplication of comparable organic-rich deposits in the  
northern and southern Tethys, in the Central Atlantic and in the Pacific. Some of these deposits could  
also have formed in shallow water anoxic conditions, which contrasts with the basin anoxia model  
characterizing some Cretaceous OAE. Anoxic conditions may have been enhanced by the conjunction  
of a contrasted arid/humid climate and the tectonic inversion of several margins. The multiplication of  
such shallow water anoxic episodes could have led to major carbon cycle perturbations from the Middle  
Oxfordian till the Early Kimmeridgian.

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## FIGURE LEGENDS

**Fig. 1:** **A)** Location of the Amu Darya and Afghan-Tajik basins in western Central Asia. ADF, Amu Darya Fault; C Ust F, Central Ustyurt Fault; STS F, South Tien Shan Fault; UKFFZ, Uchbash-Karshi Flexure Fault Zone; Za F, Zaravshan Fault. **B)** Location of the Tubiegatan section in the SW Gissar Range and geological setting of the study area (Surkhandarya province). Gau, Gaurdak section. **C-D)** View of the Khodjaipak Fm. in the Tubiegatan section (C) and of the Tubiegatan anticline (D).

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**Fig. 12:** Two correlation hypotheses between the Tubiegatan section and the south-west Gissar platform, based on the  $\delta^{13}\text{C}_{\text{carb}}$  and the sequence stratigraphy.

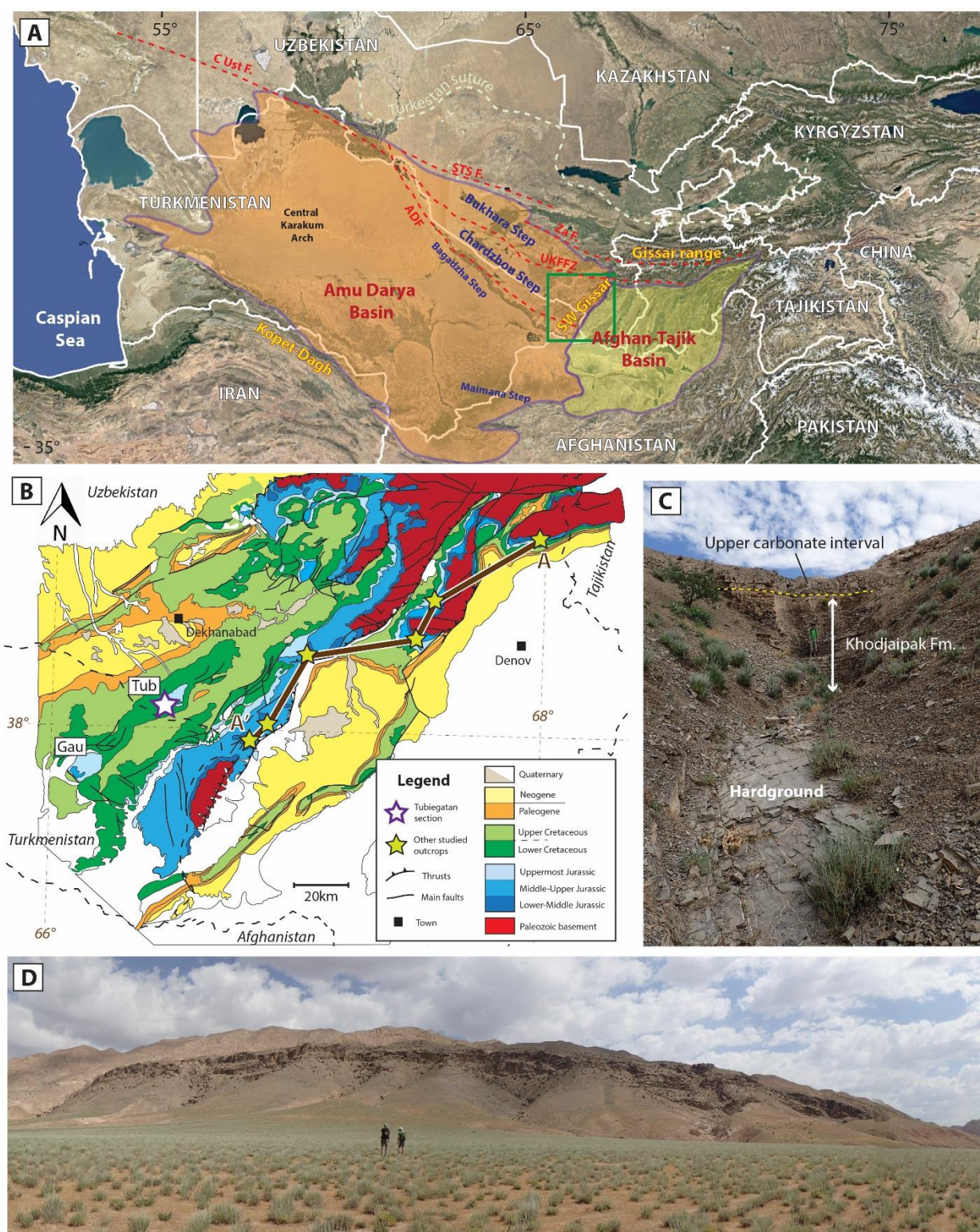
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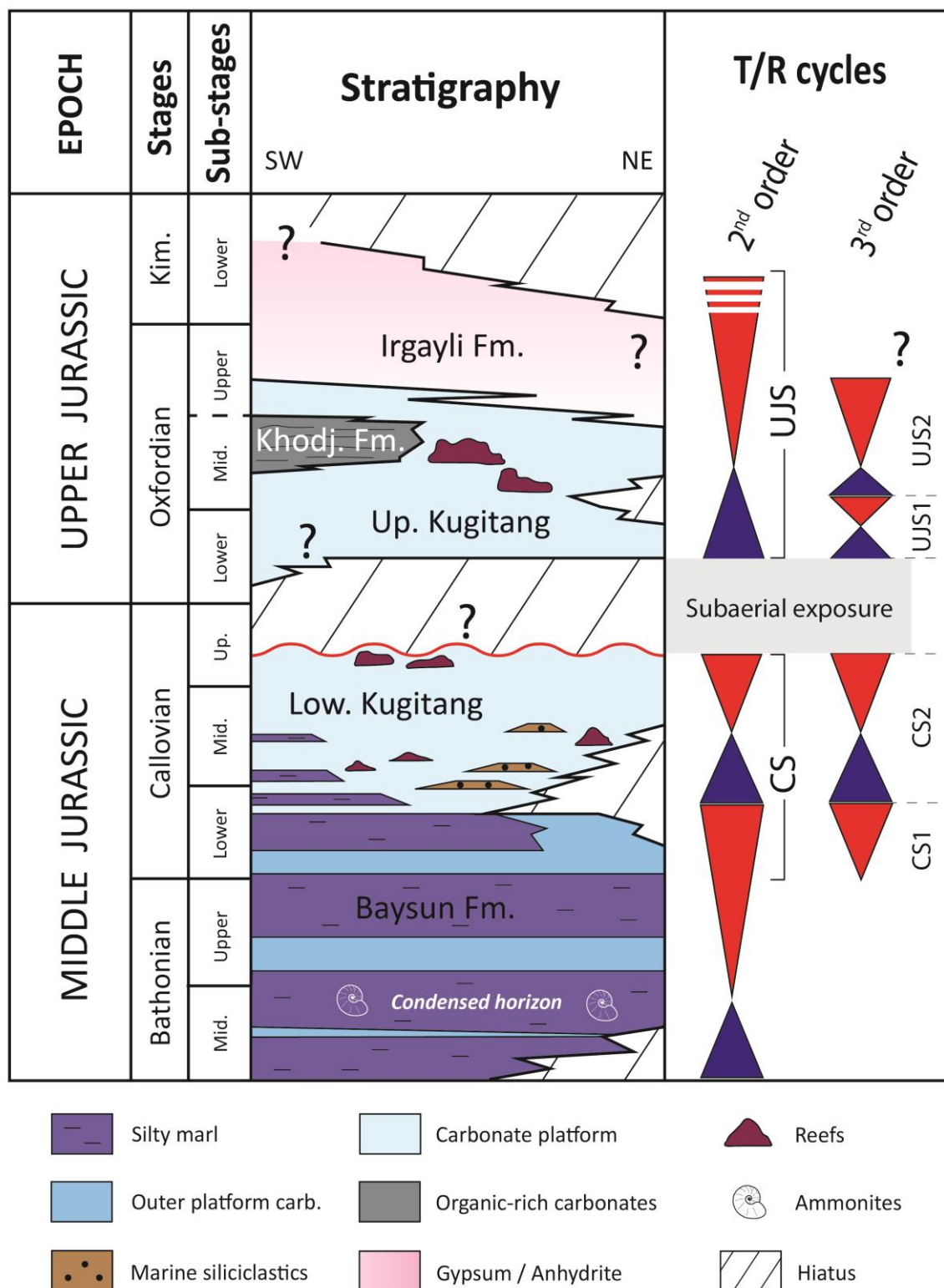
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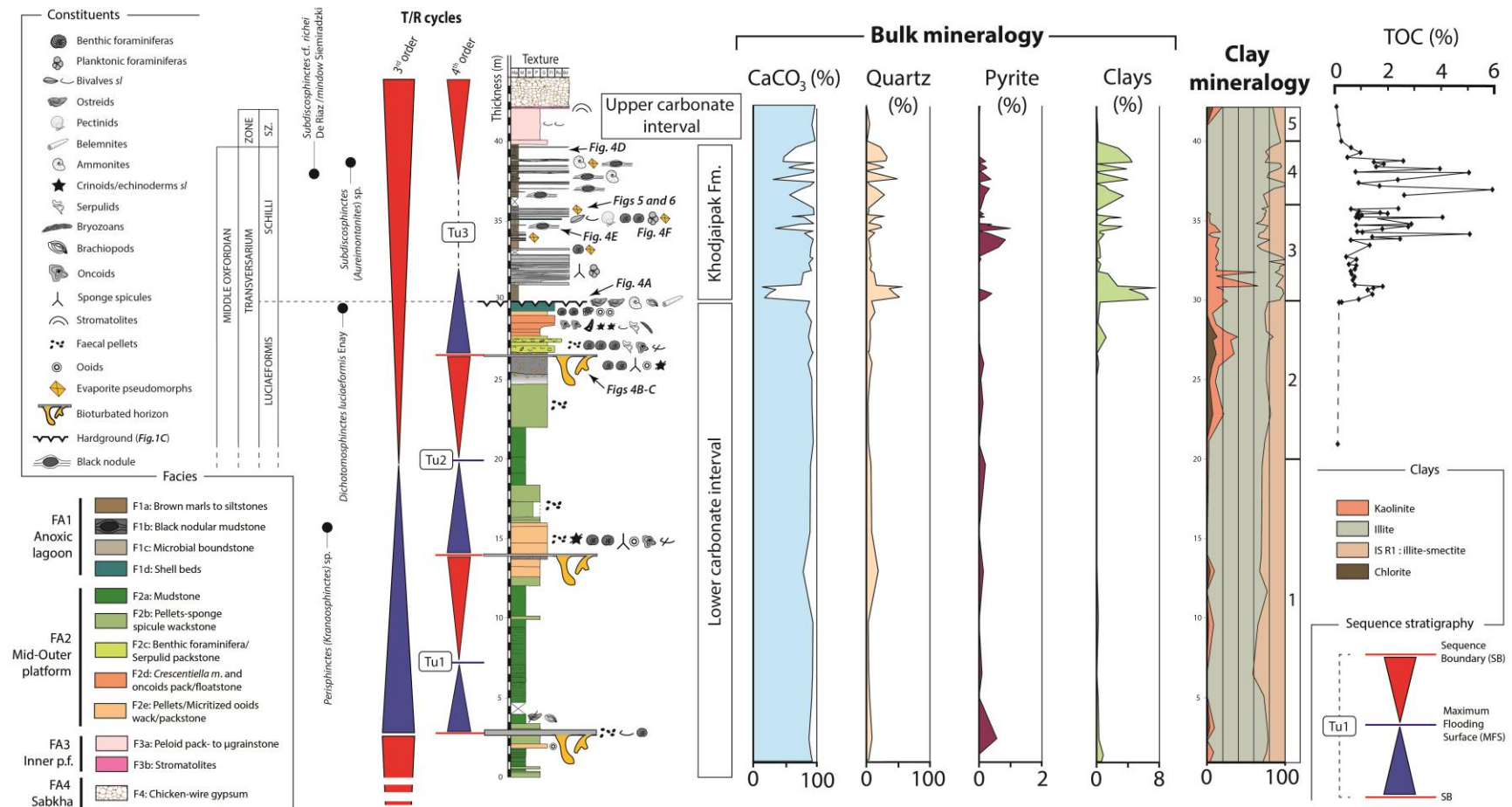


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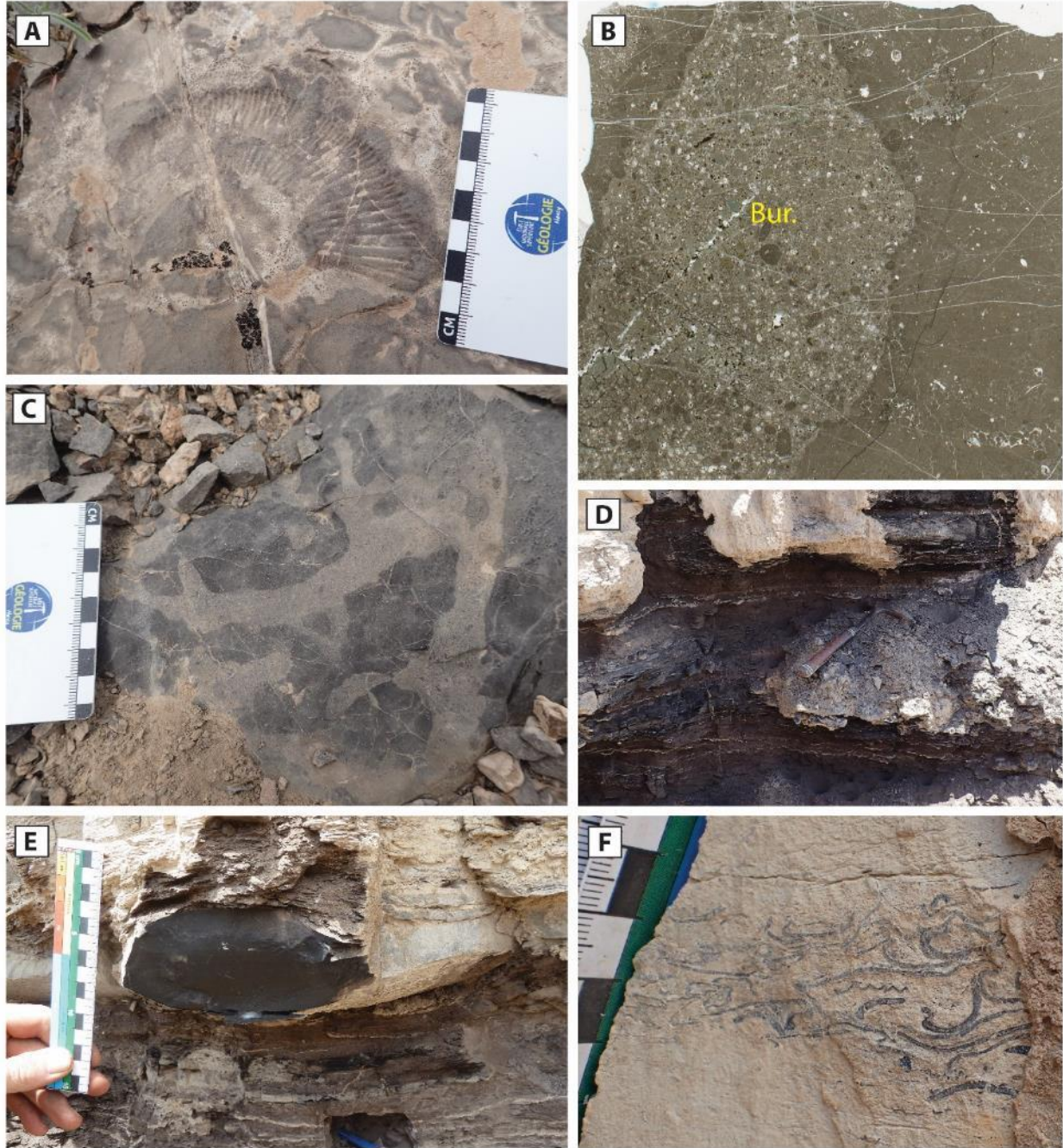


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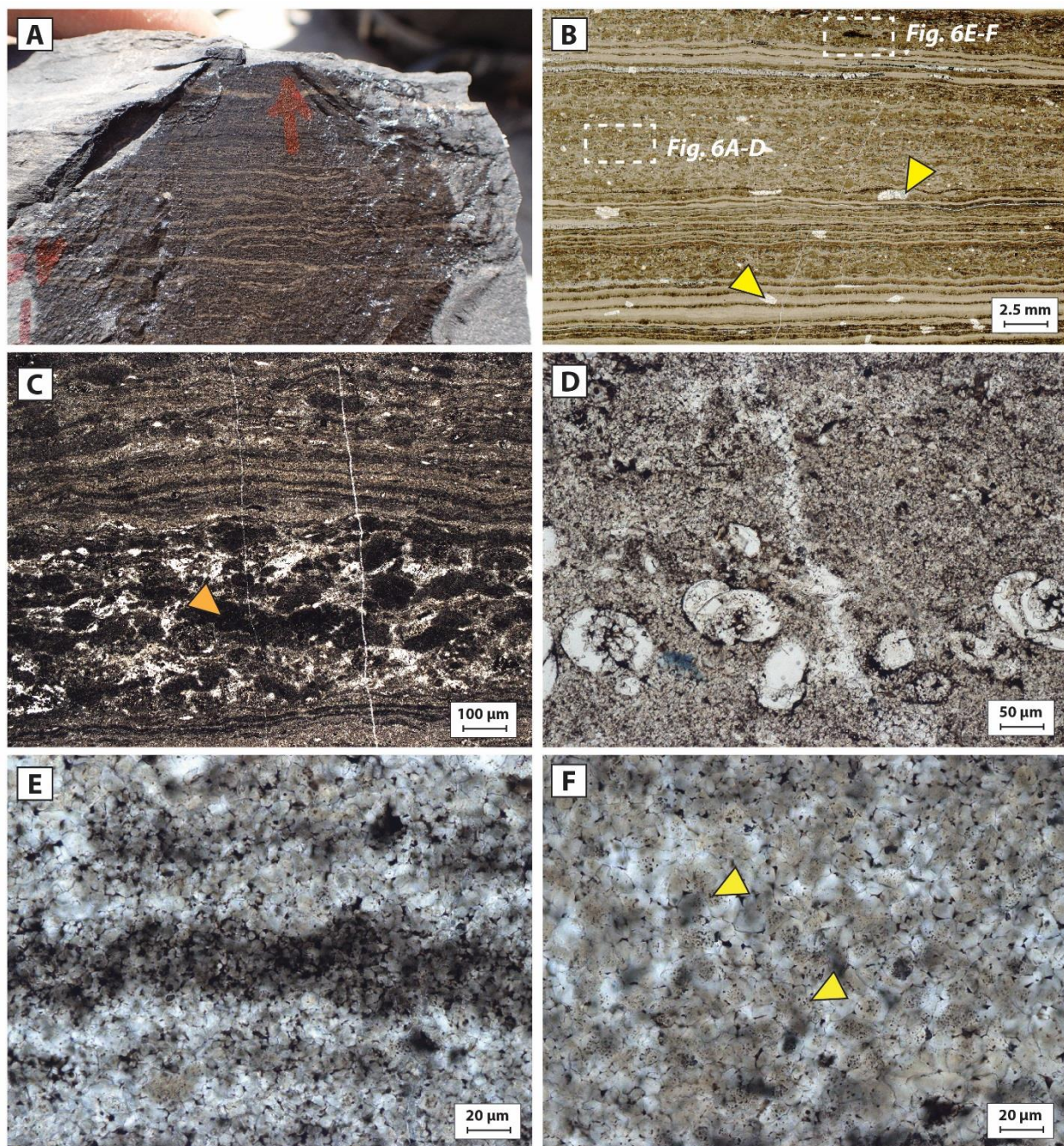
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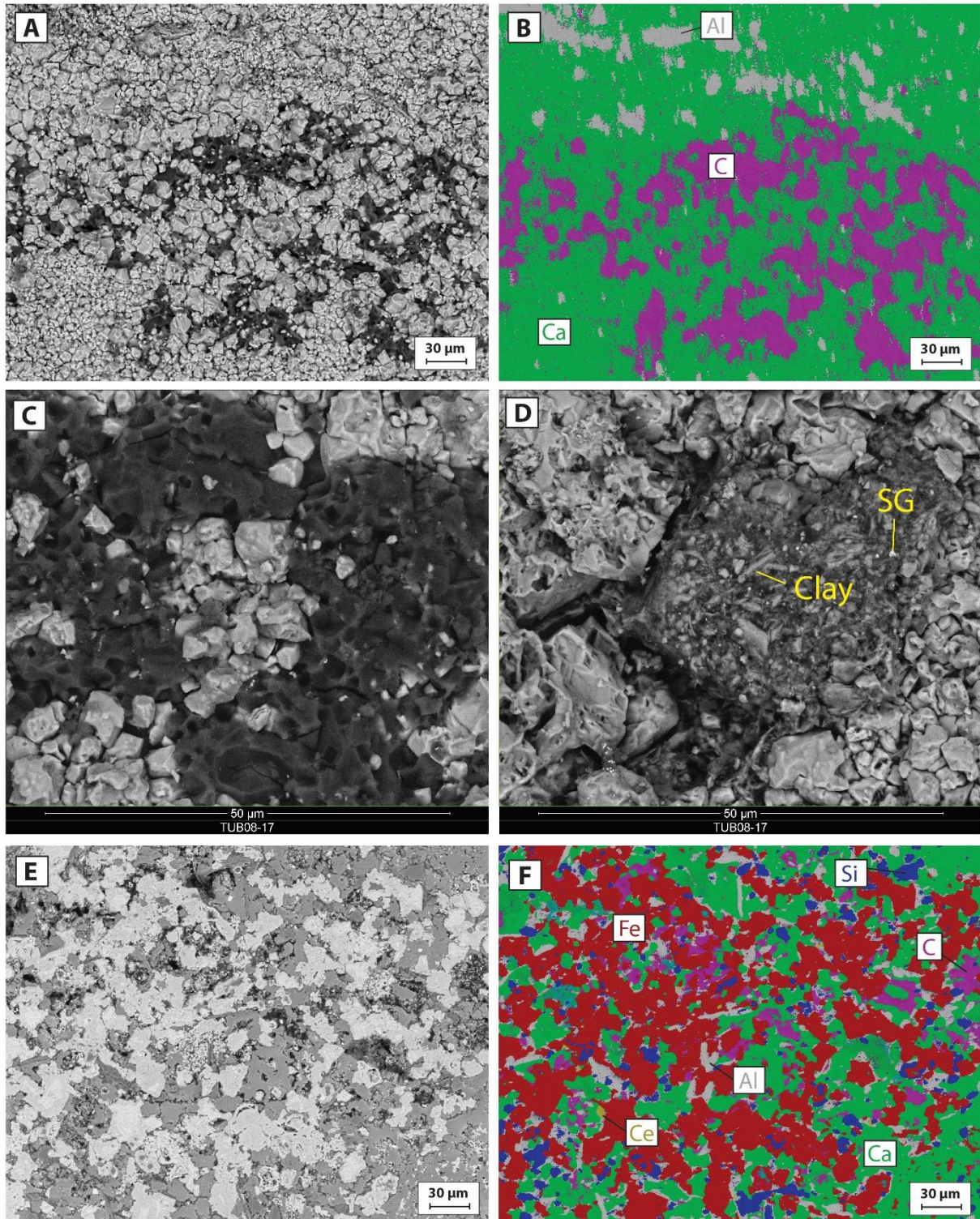
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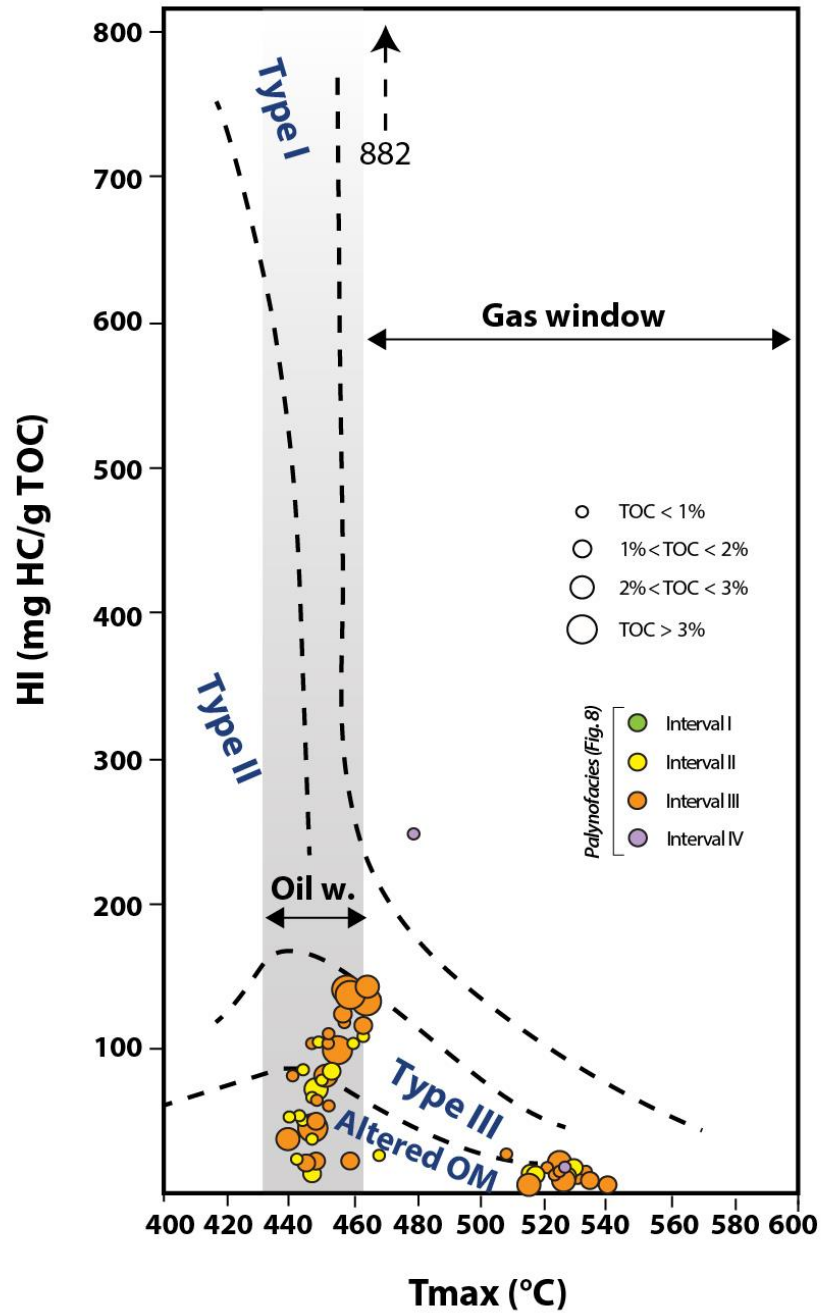
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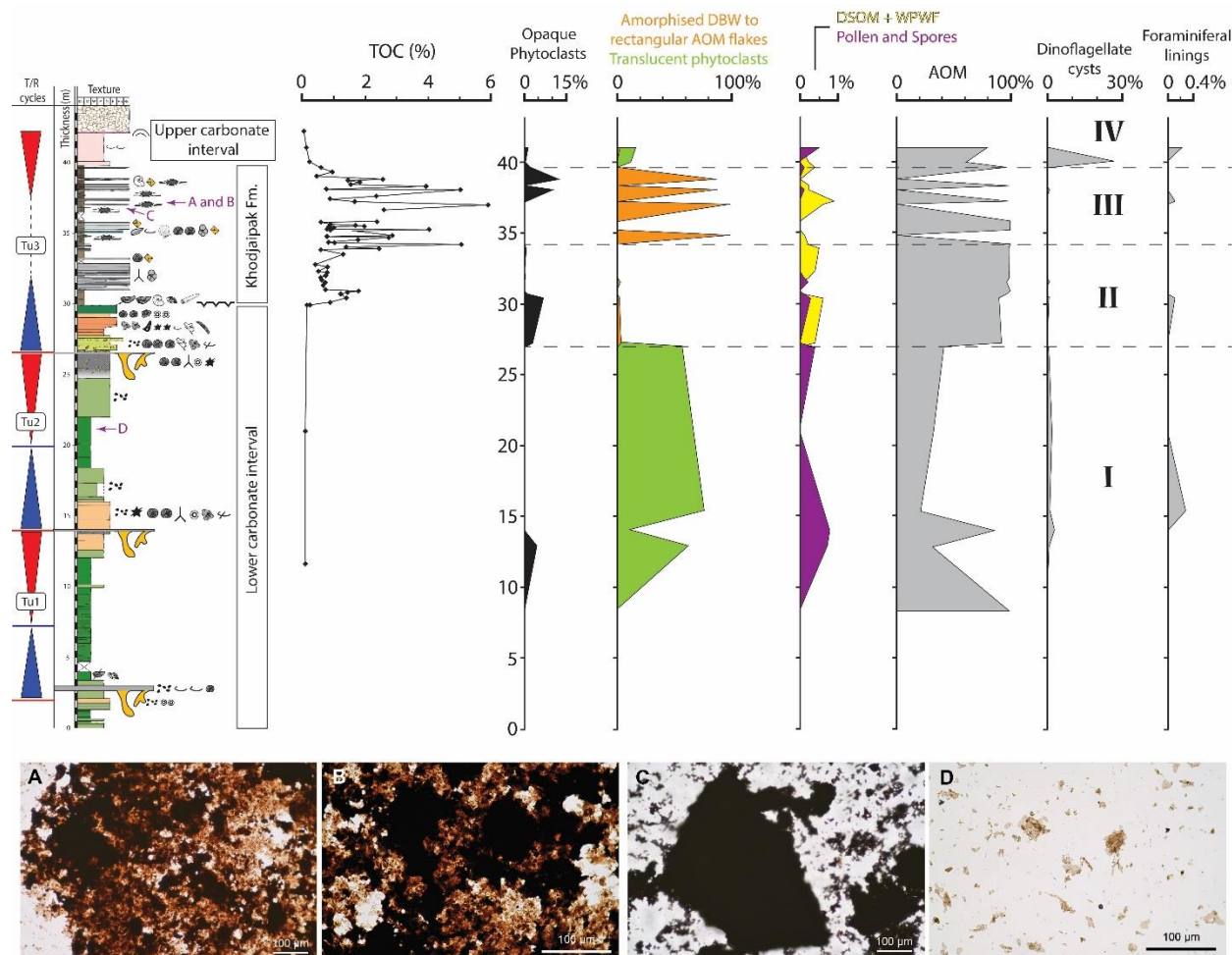


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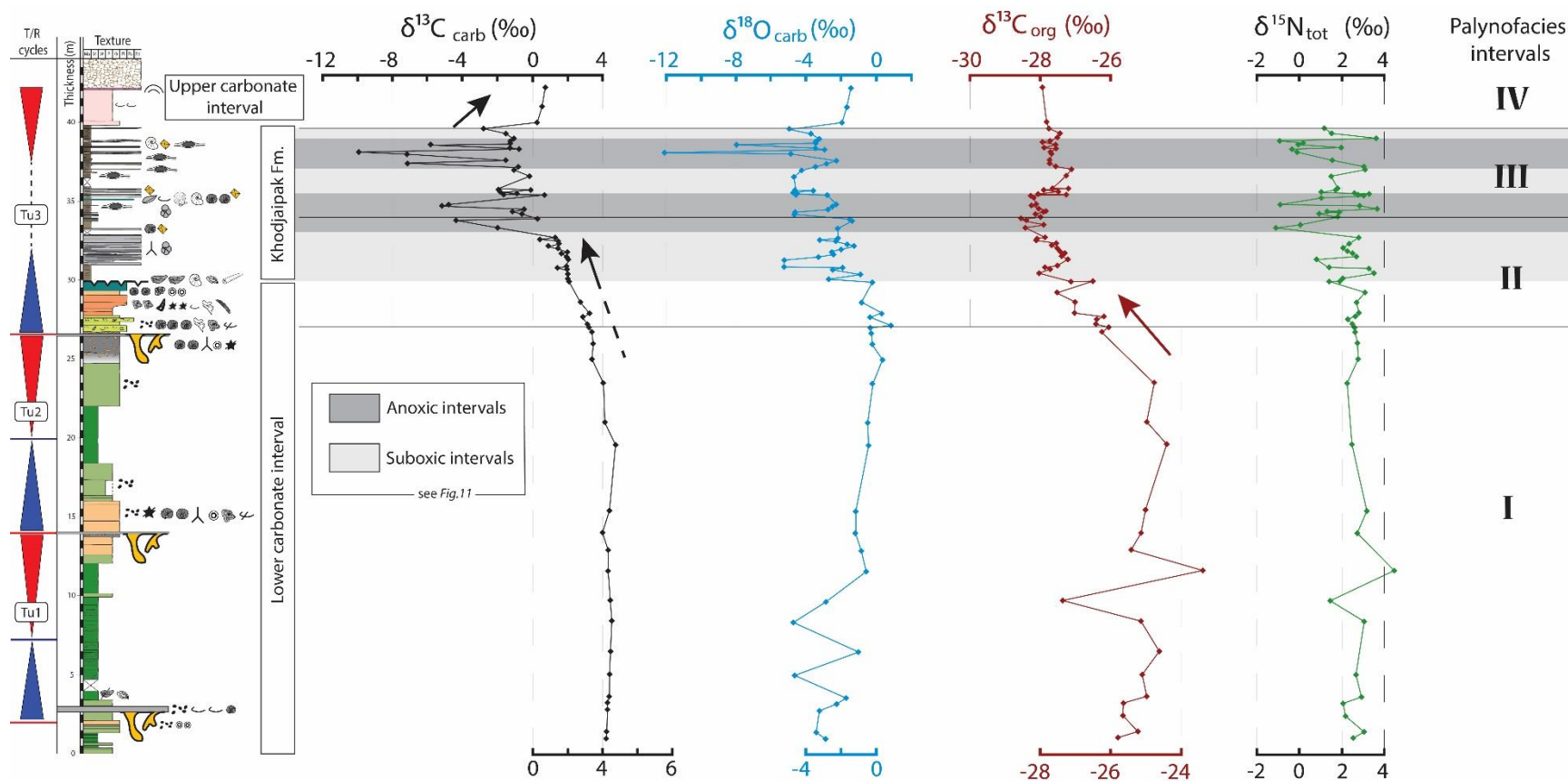




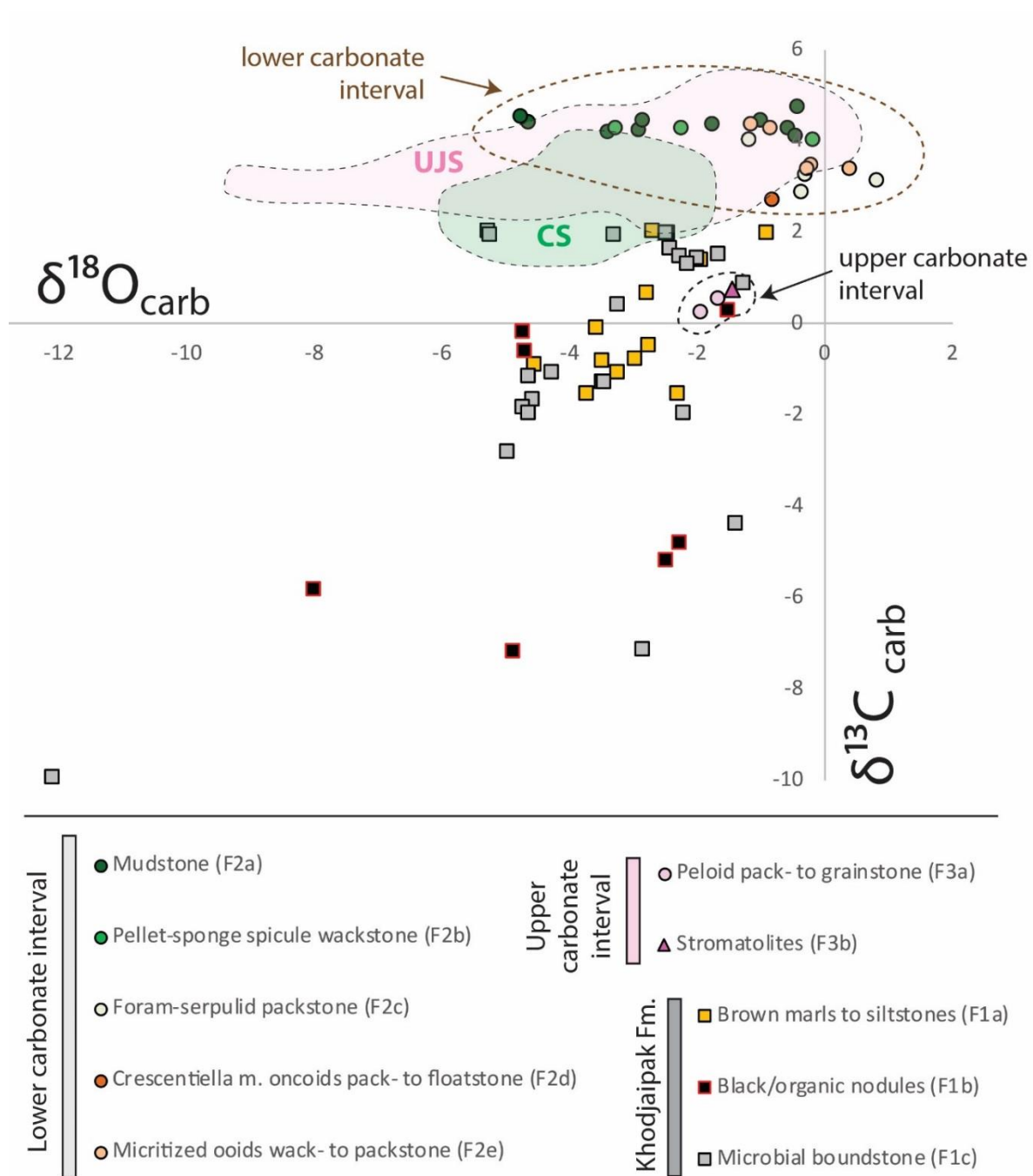
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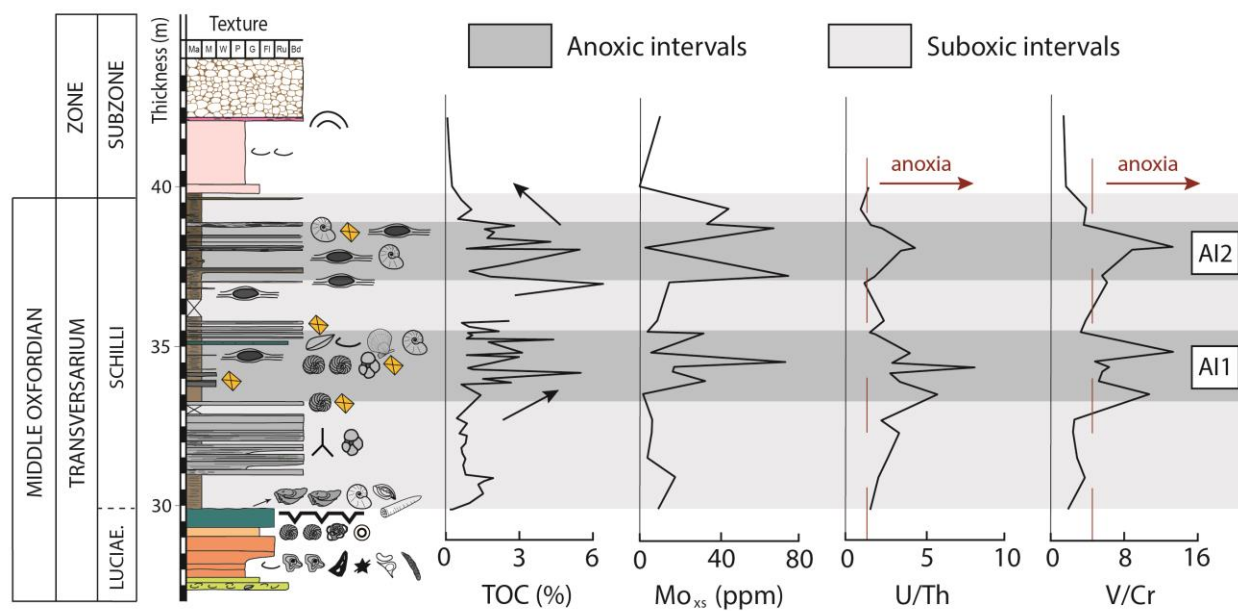
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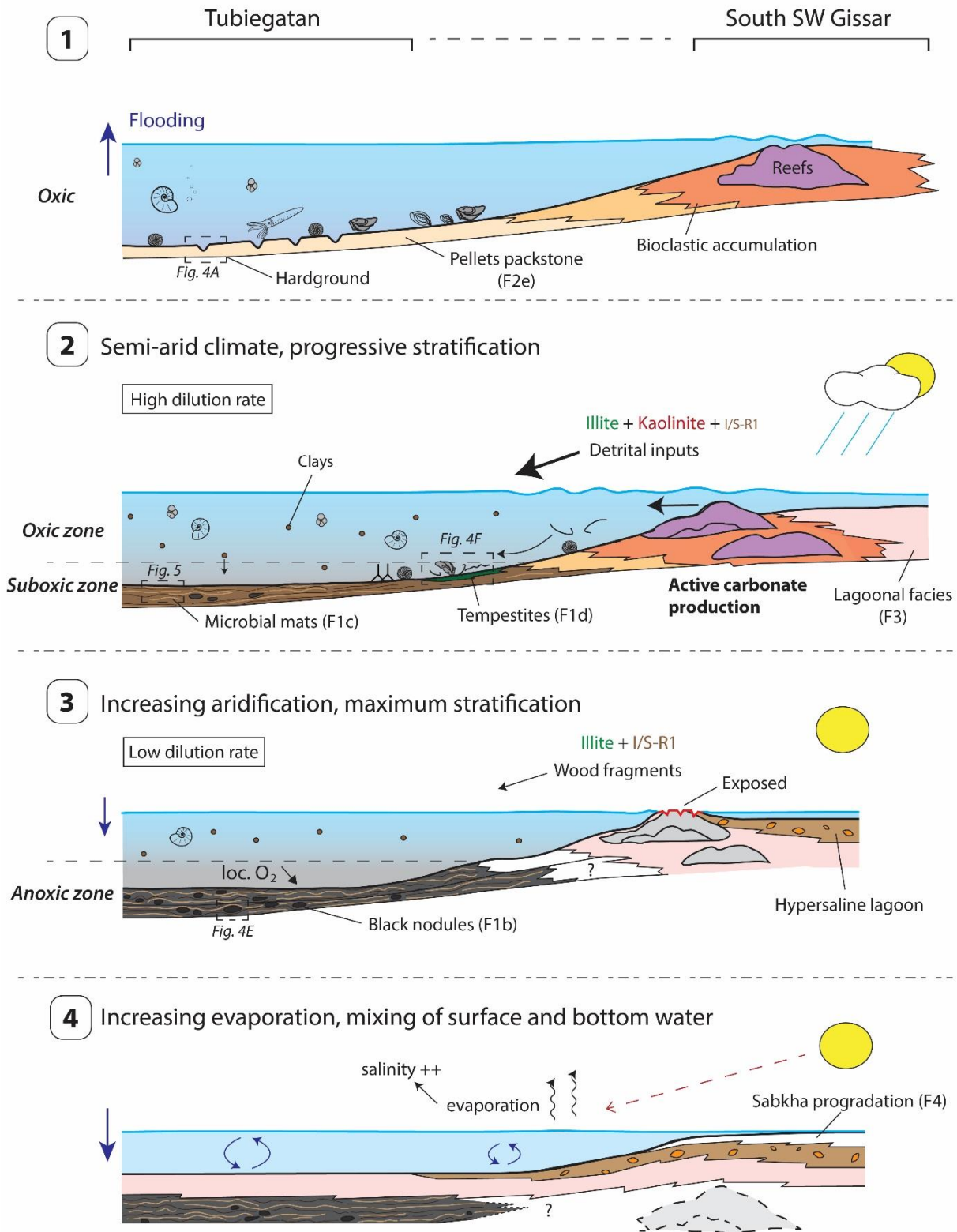


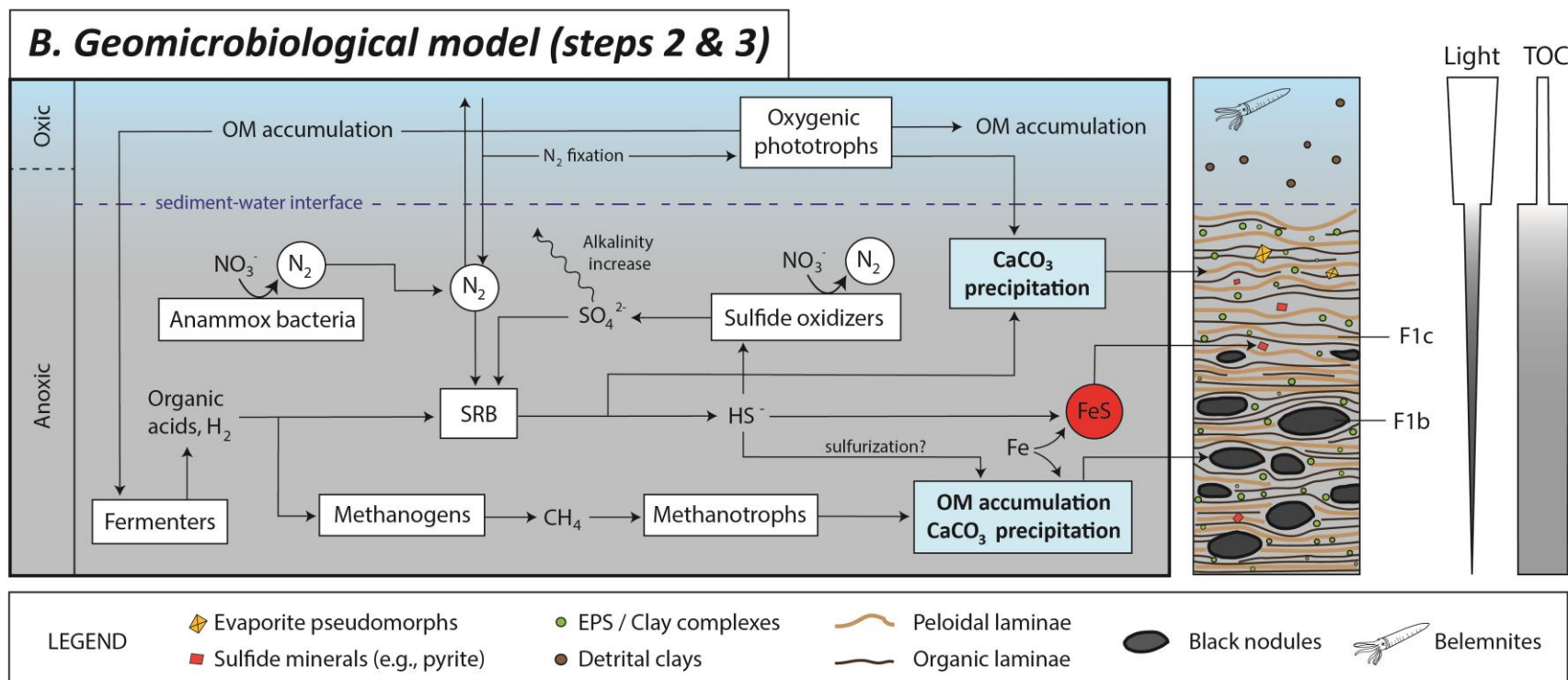
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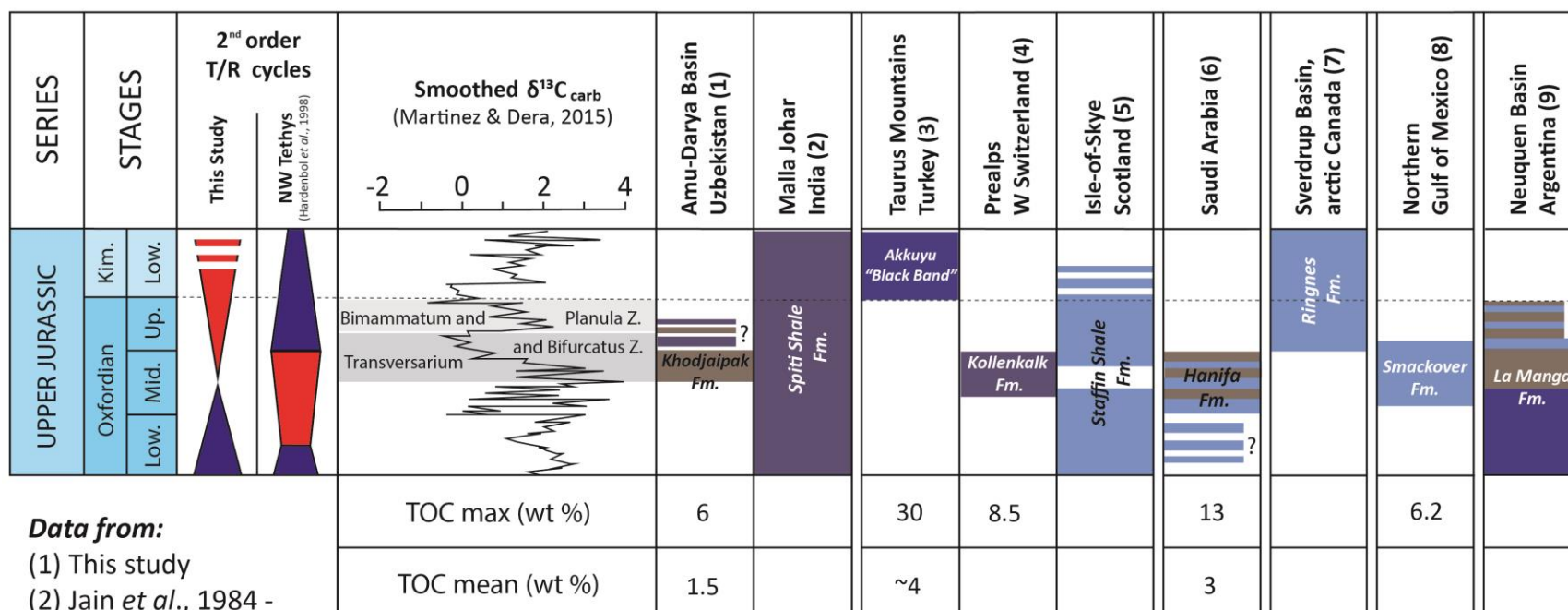
## A. Depositional scenario





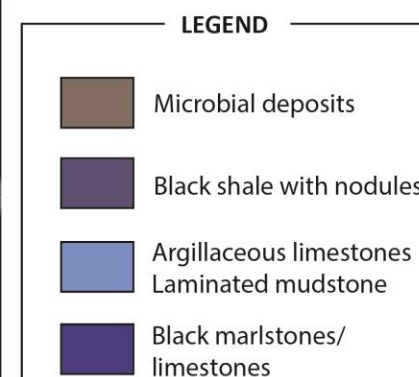
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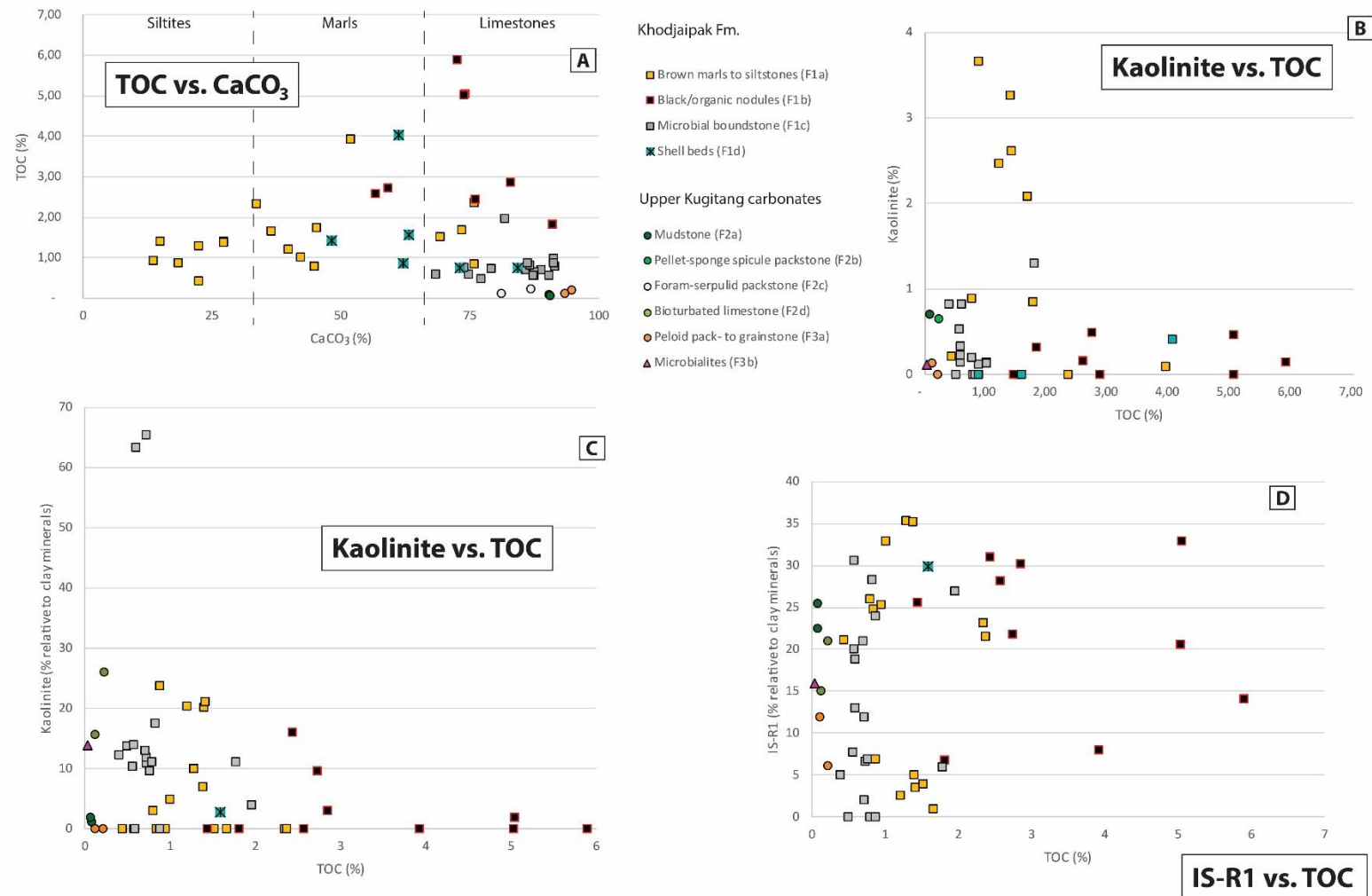


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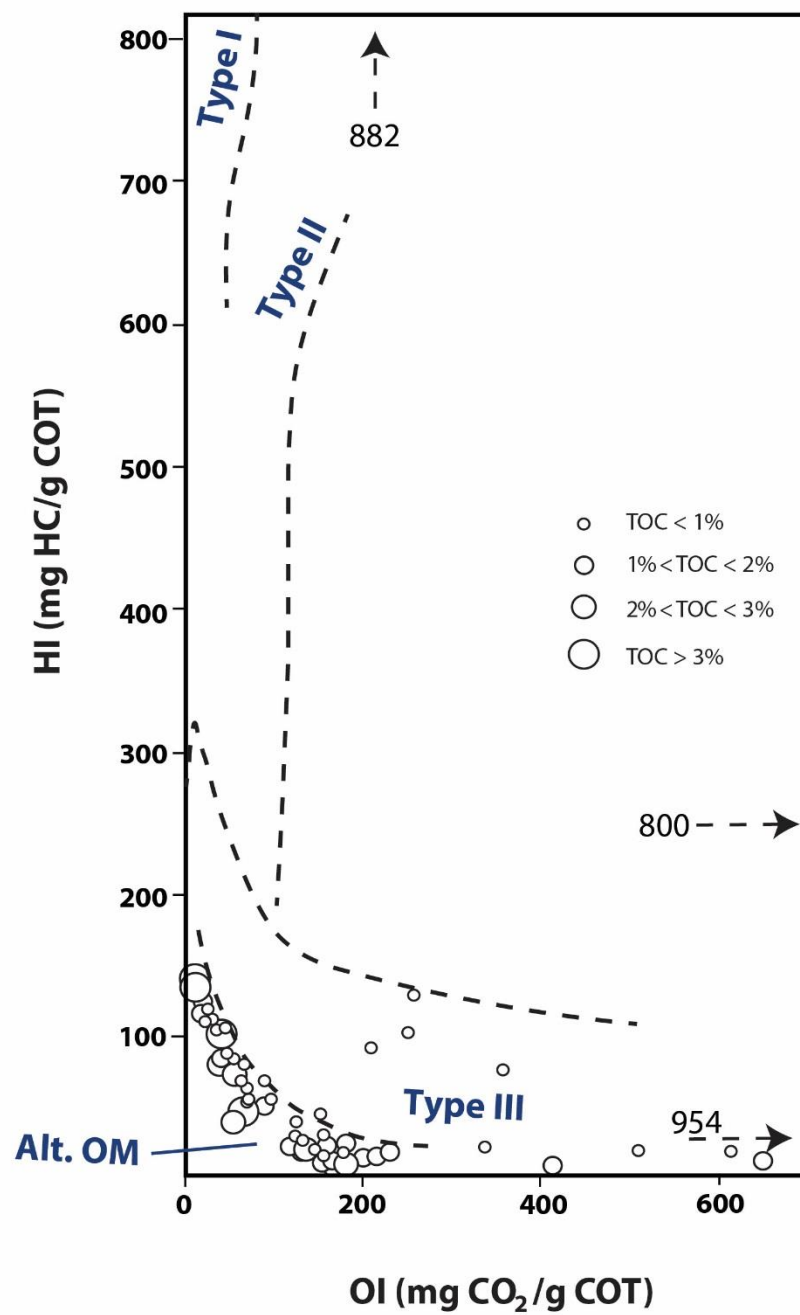
- (1) This study
- (2) Jain *et al.*, 1984 -  
Jadoul *et al.*, 1998
- (3) Baudin *et al.*, 1999
- (4) Rais *et al.*, 2007
- (5) Pearce *et al.*, 2005
- (6) Enay *et al.*, 1987 -  
Sharland *et al.*, 2001 -  
Al-Husseini *et al.*, 2006
- (7) Mukhopadhyay  
*et al.*, 1997
- (8) Heydari *et al.*, 1997  
Hammes *et al.*, 2011
- (9) Palma *et al.*, 2015



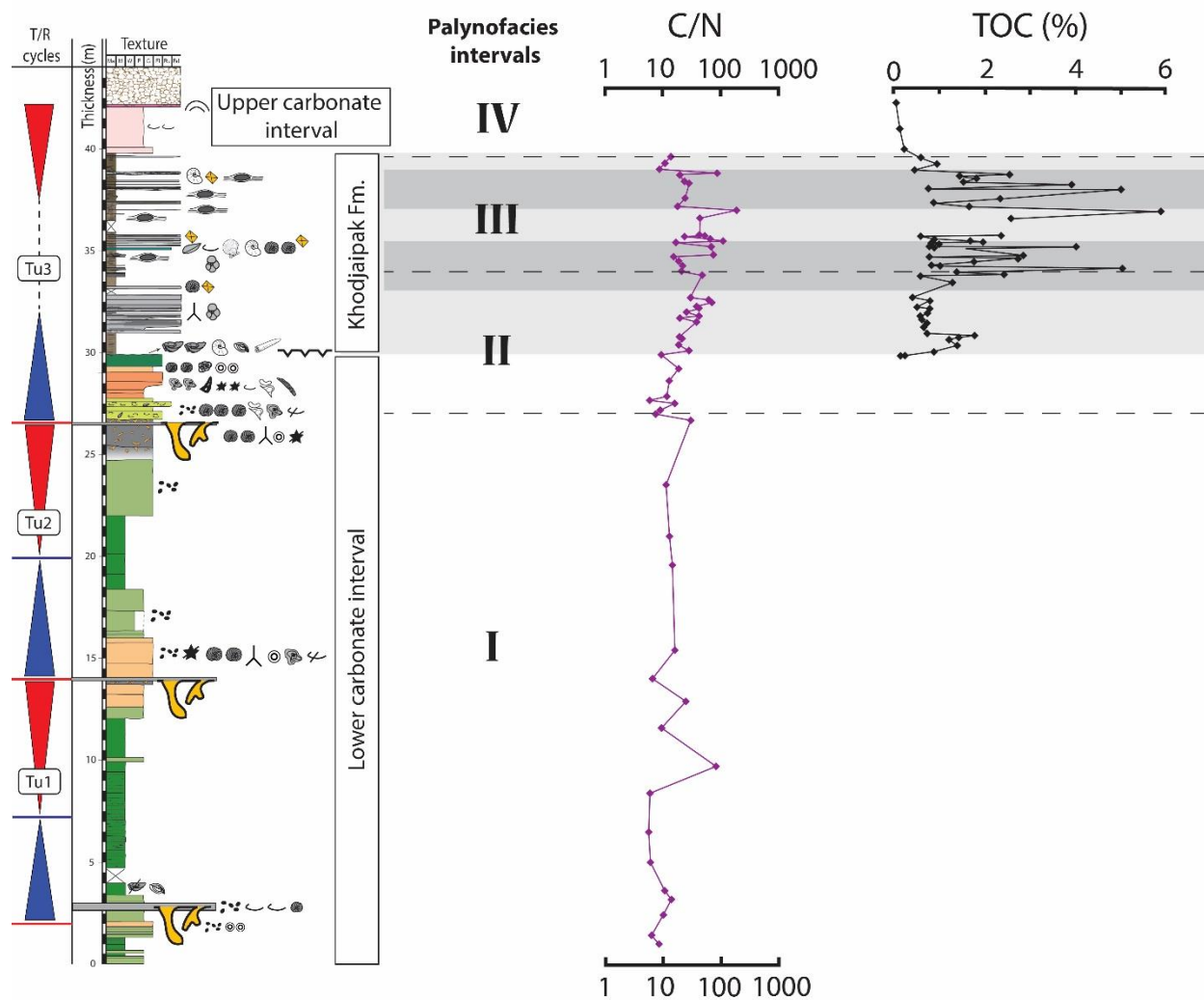
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