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Assessing controls on organic matter enrichments in hemipelagic marls of the Aptian-Lower Albian Blue Marls of the Vocontian Basin (France): an unexpected variability observed from multiple “organic-rich” levels

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Abstract – The Marnes Bleues Formation from the Vocontian Basin (Southeastern France) shows many dark-colored levels, some concomitant to oceanic anoxic events OAE1a (the so-called Goguel Level) and OAE1b (the Jacob, Kilian, and, above all, Paquier levels). These levels are usually referred to as being organic-rich; they are scattered through a thick, rather homogeneous, succession of marls, that is poor in organic matter (OM). Through a multi-parameter approach, the dark levels from the Aptian-Albian were characterized. Our results show that all dark levels are not OM-rich and that the dark levels exhibit variable characteristics, such as OM nature (marine *vs.* continental), sedimentation and accumulation rates, redox conditions, surface-water productivity and relative sea level, but they all show low to modest enrichments in OM. Furthermore, all the levels share in common the fact that they formed under conditions of normal to low productivity and oxic to suboxic conditions. Thus, our results strongly suggest that, in the absence of high productivity and anoxic bottom conditions, the other factors reputed to favor OM accumulation only led to sporadic and low enrichments in organic contents. It is as if such factors could only enhance OM accumulation but could not induce it alone. What was true for the Vocontian Basin may be extended to other settings, regardless of their time of deposition or location.

Keywords: organic matter / black shales / Marnes Bleues Formation / Cretaceous / productivity

Résumé – Évaluation des contrôles des enrichissements en matière organique dans les marnes homogènes hémipélagiques du Bassin Vocontien (France; Crétacé): une variabilité inattendue observée à partir de multiples niveaux « riches » en matière organique. La Formation de Marnes Bleues du Bassin Vocontien (sud-est de la France) présente de nombreux niveaux sombres, usuellement considérés comme étant riches en matière organique (MO), certains de ces niveaux sombres étant concomitants avec les événements anoxiques océaniques OAE1a (faisceau Goguel) et OAE1b (niveaux Jacob, Kilian, et surtout Paquier). Ces niveaux sombres sont dispersés au sein d’une épaisse succession d’apparence homogène de marnes, pauvres en MO. Grâce à une approche multi paramètres, les niveaux sombres de l’Aptien-Albien ont été détaillés. Nos résultats montrent que tous les niveaux sombres ne sont pas riches en MO et qu’ils présentent des caractéristiques variables, telles que la nature de la MO (marine *versus* continentale), les taux de sédimentation et d’accumulation, les conditions redox, la productivité de surface et le niveau relatif de la mer, mais ils montrent tous des enrichissements faibles à modestes en MO. En outre, tous les niveaux ont en commun le fait de s’être formés dans des conditions de productivité normale à faible et des conditions oxicques à suboxiques. Ainsi, nos résultats suggèrent fortement qu’en l’absence de productivité élevée et de

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conditions de fond anoxiques, les autres facteurs réputés favoriser l'accumulation de MO n'ont conduit qu'à des enrichissements sporadiques et à faibles teneurs en MO. Tout se passe comme si de tels facteurs ne pouvaient qu'améliorer l'accumulation de MO mais ne pouvaient pas l'induire à eux seuls. Ce qui était vrai pour le bassin Vocontien peut être étendu à d'autres contextes, quel que soit leur moment de dépôt ou leur localisation.

Mots clés : matière organique / schistes noirs / Marnes Bleues / Crétacé / productivité

1 Introduction

The Vocontian Basin (South-East of France; Fig. 1) accumulated the Marnes Bleues Formation (Blue Marls) during the Aptian-Albian. This formation is made of a thick succession of marls, deposited in a hemipelagic environment, displaying an apparently monotonous facies. Levels of dark shales (dark gray to black), sometimes rich in organic matter (OM), are interspersed in the Marnes Bleues: the Goguel, Niveau Noir, Fallot, Jacob Level, Kilian and Paquier levels (Bréhéret, 1997; Fig. 2). Some of these levels have lateral equivalents, identified in Italy in the Marche-Umbria Basin or in Switzerland, or at the scale of the entire Tethys (Bréhéret, 1997; Coccioni *et al.*, 2006, Stein *et al.*, 2011; Föllmi, 2012; Föllmi *et al.*, 2012; Basilone, 2021a, 2021b). Some levels correspond to oceanic anoxic events (OAE1a or OAE1b; Schlanger and Jenkyns, 1976; Arthur *et al.*, 1990; Jenkyns, 2010). Numerous studies have been devoted to these levels (Friès, 1987; Rubino, 1989; Tribovillard, 1989; Tribovillard and Gorin, 1991; Bréhéret, 1994, 1997; Friedrich *et al.*, 2003; Friès and Parize, 2003; Herrle *et al.*, 2003, 2004, 2010; Heimhofer *et al.*, 2004, 2006; Stein *et al.*, 2011; Westermann *et al.*, 2013; Ando *et al.*, 2017; Ferry, 2017; Caillaud *et al.*, 2020). In this paper, we wish to draw up an inventory of the factors involved in the deposition of these dark levels and their enrichment in OM. The question here is whether these levels always obeyed the same determinisms, among the various factors known to favor the accumulation of OM. We therefore examined the OM content of the sediments and the parameters of Rock-Eval pyrolysis, the grain size distribution of the carbonate-free fraction, the composition of the clay mineral assemblage, the concentrations of major and trace elements, the position of the levels rich in OM in a sequence stratigraphy framework, and sedimentation and accumulation rates were calculated. Finally, the question is also to know if the Aptian/Albian of the Vocontian Basin was a specific case or if the lessons learned from this study have a more general value.

2 Geological setting of the Vocontian Basin during the Lower Cretaceous

The South-East Basin of France corresponds to what was the slope of the northern margin of the Tethys Ocean during the Lower Cretaceous (Friès and Parize, 2003 and references therein, Figs. 1A and 1B). Thus, the Vocontian Basin was a sort of diverticulum or large gulf of the Tethys, only connected with the open ocean through its east end. A system of extensive faults and tilted blocks, inherited from the Tethyan rifting during the Jurassic times, controlled the geometry of the basin

(Graciansky and Lemoine, 1988). Then, the transition to a coupled extensive-transpressive tectonic system took place during the Aptian-Albian period (Beaudoin *et al.*, 1986; Friès, 1987; Graciansky and Lemoine, 1988; Joseph *et al.*, 1989). Depending on the localization in the Vocontian Basin, this structural control resulted in contrasting thickness of sediment successions.

3 The Marnes Bleues Formation

In the Vocontian Basin, during the Aptian-Albian period, a clastic-dominated sedimentation took place (Friès, 1987; Rubino, 1989; Bréhéret, 1997). In the deep part of the basin, the marl-dominated hemipelagic facies of the Marnes Bleues Formation (Fm.) accumulated above the Barremian/lower Aptian carbonates (Friès, 1987; Fig. 2A). A global "carbonate crisis" could explain this lithological change (Cotillon *et al.*, 2000 and references therein). The Marnes Bleues Fm. is mainly composed of dark-grey marls with low OM contents (0.5% on average), with several occurrences of organic-rich levels (TOC > 1.5%) and some carbonate horizons (Tribovillard, 1989; Bréhéret, 1997). The mean carbonate content in the Marnes Bleues Fm. is comprised between 25% and 35%. There are also numerous deposits, like slumps, massive sandstones, turbidites, and debris-flows, mainly localized in the NW part of the Vocontian Basin (Friès, 1987, Rubino, 1989; Friès and Parize, 2003). The thickness of the entire Marnes Bleues Fm. is comprised between 650 and 800 m (Friès, 1987; Bréhéret, 1997), and the Aptian part is about 300-m thick. The biostratigraphic divisions were established after ammonite and foraminifer identification (Friès, 1987; Bréhéret, 1997; Dauphin, 2002). The rocks of the Marnes Bleues Fm. contain 30% to 50% of clay-sized particles (< 2 μm ; Friès and Parize, 2003). Illite, smectite, and illite-smectite mixed-layers minerals dominate the clay mineral distribution of the terrigenous fraction.

The paleodepth was estimated to approximately 100 m on the platforms (Arnaud-Vanneau and Arnaud, 1991), and between 500 to 1500 m on the ancient slope of the margin (Guérin, 1981; Beaudoin *et al.* 1986; Arnaud-Vanneau and Arnaud, 1991), according to the (planktonic and benthic) foraminifer studies and the estimations deduced from the geometry of reworked deposit.

The sequence-stratigraphy framework highlights five third-order depositional sequences from the Aptian to the Lower Albian (Fig. 3; Rubino, 1989; Friès and Parize, 2003; Bréhéret, 1997; Ferry, 2017). Despite small diverging interpretations, all studies agree with the global regressive trend recorded through these depositional sequences, highlighting the progressive closure of the Vocontian Basin during the mid-Cretaceous period.

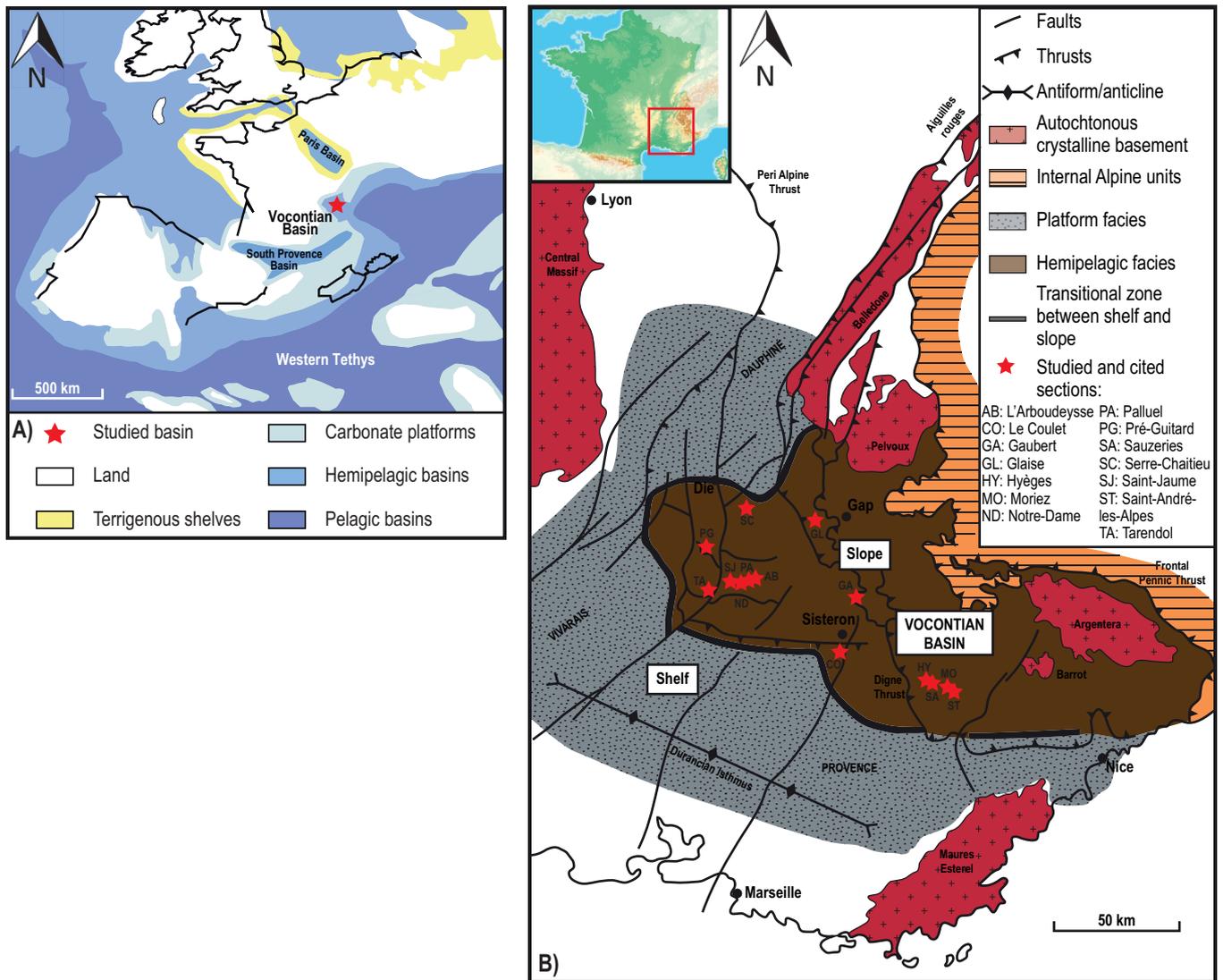


Fig. 1. (A) Aptian paleogeographical map of the western Tethys (after Stein *et al.*, 2011, modified from Masse *et al.*, 2000). (B) Map of the geological context of the Vocontian Basin during the Aptian times. Modified from Friès and Parize (2003).

Fig. 1. (A) Carte paléogéographique de la Téthys occidentale aptienne (d'après Stein *et al.*, 2011, modifiée d'après Masse *et al.*, 2000). (B) Carte du contexte géologique du Bassin Vocontien à l'Aptien. Modifié d'après Friès et Parize (2003).

4 Dark levels within the Marnes Bleues Formation

The Marnes Bleues Fm. composed of grey marls, shows various dark horizons, sometimes structured in paired or triple, thin, black horizons. The black horizons of the Vocontian Basin are frequently termed organic-rich levels in the literature (but we will see herein below that it is not always true); for instance, the Goguel and Paquier levels display a facies that may be termed black shale, but the other dark levels stand out of their host marls only thanks to their darker coloration. Table 1 summarizes the main features of these organic-rich levels.

5 Materials and methods

5.1 Sampling and sections

One hundred and sixty-five samples were selected from 10 sections:

Goguel Level (32 samples labeled with “-g” termination), Niveau Noir Interval (4 samples labeled with “-n” termination), Fallot Interval (21 samples labeled with “-f” termination), Jacob Level (3 samples labeled with “-j” termination), Kilian Level (2 samples labeled with “-k” termination), Paquier Level (8 samples labeled with “-p” termination), Hemipelagites – corresponding to the background marly sedimentation (62 samples labeled with “-ah” termination),

For each sample, 500 g to 1 kg of rock were sampled. To avoid any contamination by hand skin, plastic or cardboard, the rock samples were kept in aluminum foils. The studied sections are discussed in the section 4, and they are represented on the composite type-section of the Marnes Bleues Fm. (Fig. 2A). For more details about the section locations and the references used, see Table S1.

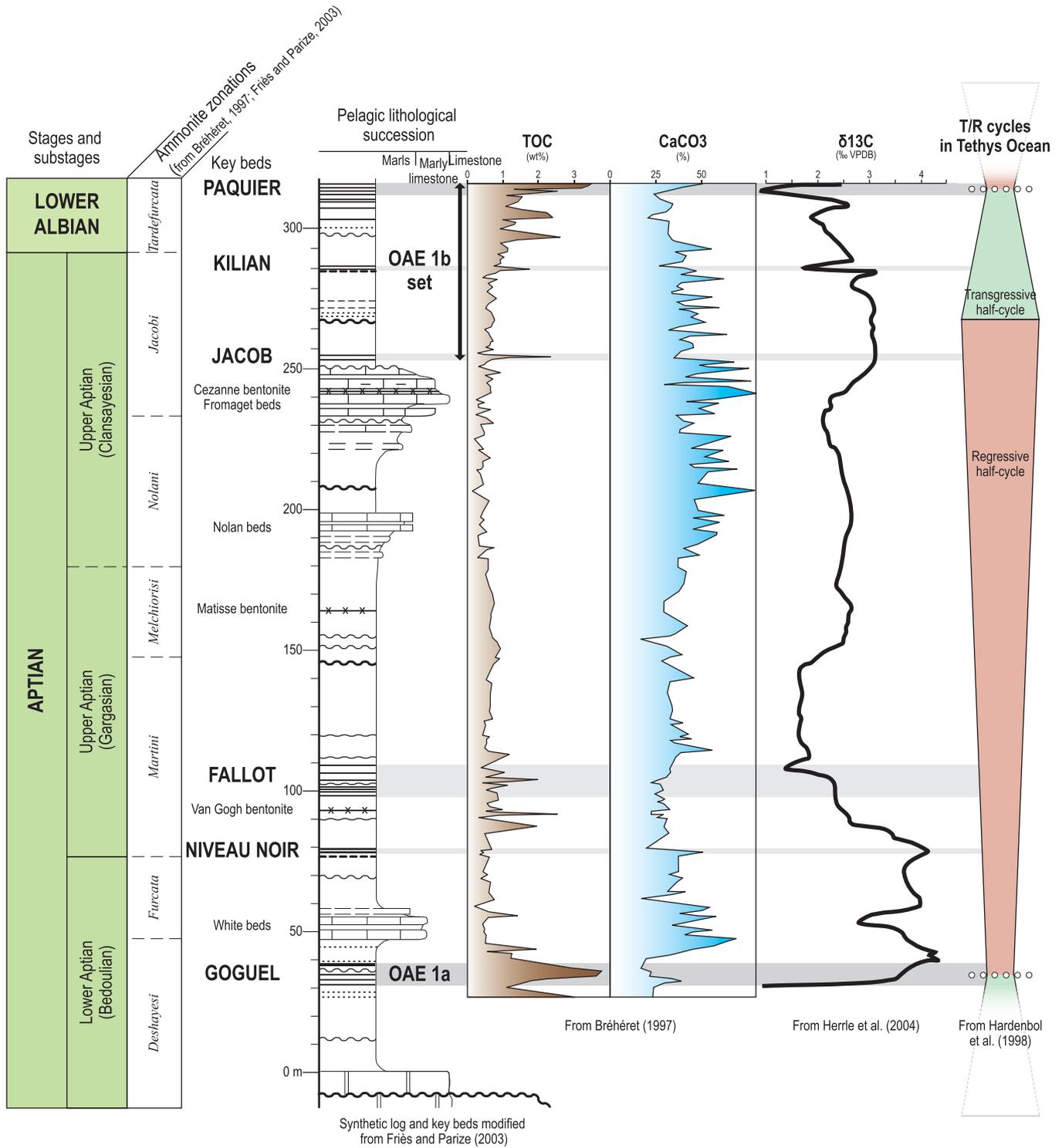


Fig. 2. Synthesis of lithology, biostratigraphy and sequence stratigraphy for the Marnes Bleues Fm. (A) Synthesis of organic and carbonate contents, stable isotope of carbon ($\delta^{13}\text{C}$), and transgressive/regressive cycles during the Marnes Bleues Fm. deposition.

Fig. 2. Synthèse de la lithologie, biostratigraphie et stratigraphie séquentielle pour les Marnes Bleues. (A) Synthèse des teneurs organiques et carbonatées, isotopes stables du carbone ($\delta^{13}\text{C}$), et cycles transgressifs/régressifs au cours du dépôt des Marnes Bleues.

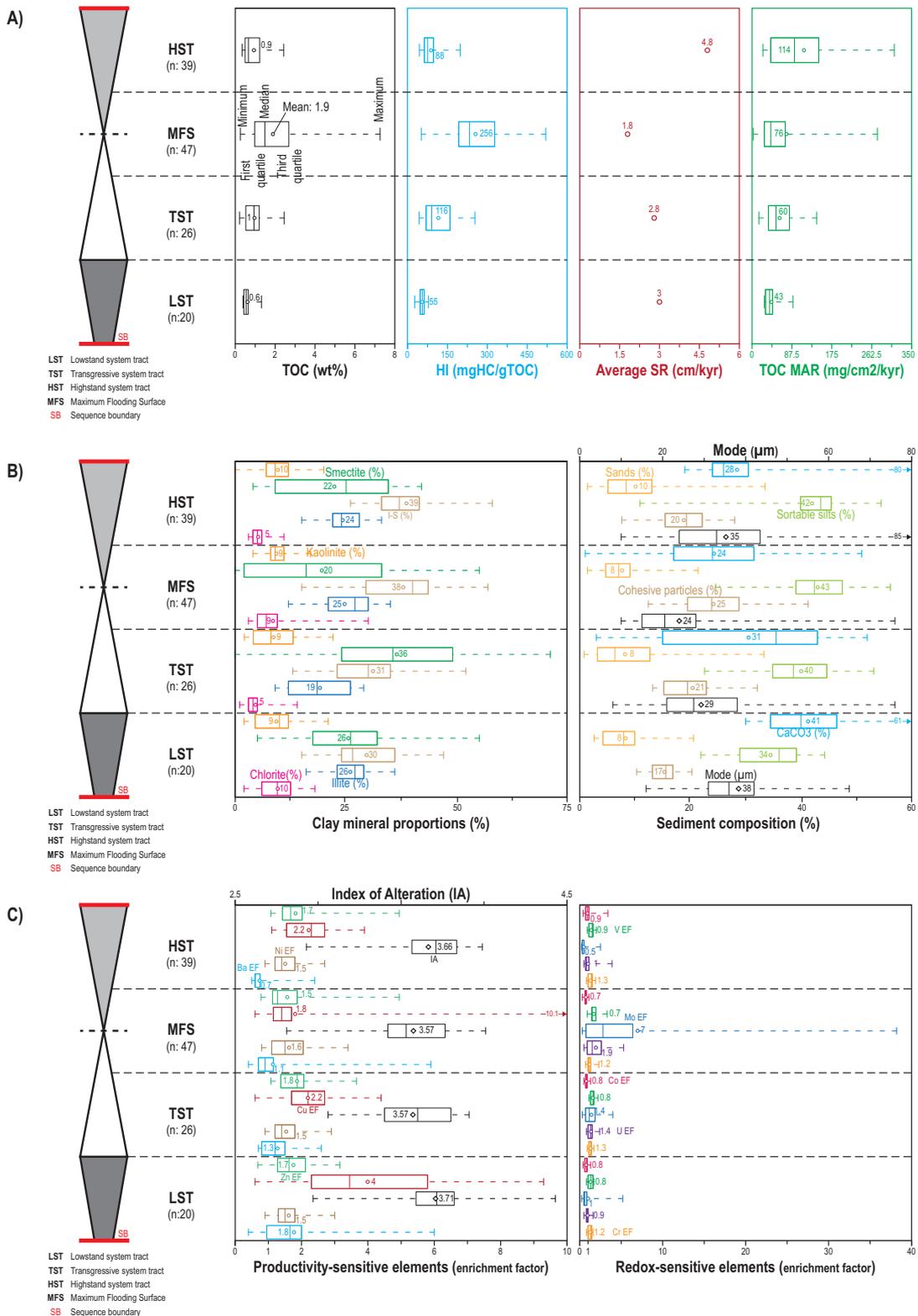


Fig. 3. Clay mineral proportion, sediment composition and grain size mode: (A) within the sequence-stratigraphy framework; and, (B) in the Aptian to Lower Albian interval. organic-rich levels display on the type section are not at scale.

Fig. 3. Proportion de minéraux argileux, composition des sédiments et mode granulométrique : (A) dans le cadre de la stratigraphie séquentielle ; et, (B) dans l'intervalle de l'Aptien à l'Albien inférieur. Les niveaux riches en MO affichés dans la section de type ne sont pas à l'échelle.

Table 1. Descriptions of the various dark levels.**Tableau 1.** Descriptions des différents niveaux riches en MO.

Organic-Rich Levels (OL)	Description
The Goguel Level	The Goguel Level is made up with six horizons of dark, marly, laminated shales (GO 1 to GO 6) amounting to a thickness of about 3.4 m in the Sauzeries section, with each horizon being multi-cm to multi-dm thick (Bréhéret, 1997). However, the laminated horizons are often interbedded with thin turbidites in numerous locations (Friès, 1987; Bréhéret, 1997; Friès and Parize, 2003; Caillaud <i>et al.</i> , 2020). For example, the Goguel Level reaches a thickness of 17 m in the Saint-Jaume section, where it is interbedded with thin turbidites and one slump. This OL records a firstly negative and then positive $\delta^{13}\text{C}$ excursion in the Vocontian Basin (Herrle <i>et al.</i> , 2004; Westermann <i>et al.</i> , 2013), as is the case for the OAE1a in the Tethyan basins (Menegatti <i>et al.</i> , 1998). The Goguel Level contains a total organic carbon (TOC) of 1 to 5 wt% with hydrogen index (HI) values up to 500 mgHC/gTOC, which corresponds to Type-II kerogens, dominated by marine algal-bacterial OM (Bréhéret, 1994, 1997; Heimhofer <i>et al.</i> , 2004, 2006; Westermann <i>et al.</i> , 2013; Ando <i>et al.</i> , 2017; Caillaud <i>et al.</i> , 2020). This level was the time-equivalent of the OAE1a (Bréhéret, 1994, 1997) and, therefore, it has many equivalent levels in other basins: the Livello Selli in Italian basins, the Fischschiefer in the Lower Saxony Basin, and some other black shales in the Tethyan basins and Atlantic and Pacific Oceans (Föllmi, 2012). The astronomical calibrations allowed to determine a duration of ~ 1.1 Ma for the OAE1a (Malinverno <i>et al.</i> , 2010). The Goguel Level was deposited during a major transgression recorded in the Tethys Ocean (Haq <i>et al.</i> , 1987; Hardenbol <i>et al.</i> , 1998), and coincided with a MFS (Rubino, 1989; pers. com. in 2017; Bréhéret, 1997) or even a “flash” transgression in the Vocontian Basin (Ferry, 2017). In the Tethyan realm, the deposition of the black shales was accounted for by intense surface-water productivity at the onset of the OAE1a (Weissert, 1990; Föllmi, 1995; Sanfourche and Baudin, 2001; Westermann <i>et al.</i> , 2013). In the Vocontian Basin, the model was more specific: the Goguel Level was deposited during a period of sedimentary condensation (Bréhéret, 1997; Heimhofer <i>et al.</i> , 2006) in a stratified basin (Caillaud <i>et al.</i> , 2020), but, the efficient preservation of OM was depending on the possible occurrence of thin turbidites. Therefore, where thin turbidites were interbedded within the Goguel Level, they favored dilution and remineralization of OM, whereas where the Goguel Level was deposited without turbidites, the concentration and preservation of OM was furthered (see Caillaud <i>et al.</i> , 2020 for details).
The Niveau Noir Interval	The Niveau Noir Interval underlines a basin-wide color change from pale to dark marlstones (Rubino, 1989). Over about 8 m in the Saint-Jaume section, this interval is composed of one pair of dark marly shale horizons and three pairs of slightly lesser dark marly shale beds (NN1 to NN4, Bréhéret, 1997) interbedded within grey marlstones. In the Vocontian Basin, this OL coincides with a positive $\delta^{13}\text{C}$ excursion (Herrle <i>et al.</i> , 2004,) and its duration was estimated to ca. 300 kyr using cyclostratigraphy (Dauphin, 2002). The Niveau Noir Interval is termed “black shales” or “organic-rich level” in the literature (Herrle <i>et al.</i> , 2003, 2010; Föllmi, 2012; Ghirardi <i>et al.</i> , 2014); however TOC contents are comprised between 0.7 and 1.2 wt% only (Bréhéret, 1997). This interval shows very low HI (< 100 mgHC/gTOC), corresponding to Type-III kerogens (terrestrial or degraded OM). The Niveau Noir Interval was deposited at a 3rd-order MFS (Rubino, 1989; Friès and Parize, 2003) and was ascribed the “productivity-driven model” by Herrle <i>et al.</i> (2010). This model involves a period of strong surface-water productivity and evaporation, enhancing oxygen depletion in the deep basin and therefore the establishment of a water-column stratification (based on a study of calcareous nannofossil, Herrle <i>et al.</i> , 2010).
The Fallot Level	Throughout an interval of about 21 m in the Serre-Chaitieu section, the Fallot Interval corresponds to numerous bundles of two or three horizons each (FA 1 to FA 4, Bréhéret, 1997) of dark marlstones within grey marlstones, with multi-cm to m thickness for each bundle (Bréhéret, 1997, Dauphin, 2002, Friedrich <i>et al.</i> , 2003). Based on astronomical calibration performed on the Piobbico core in Central Italy, the duration of the Fallot Interval was estimated to be ~ 360 kyr (Huang <i>et al.</i> , 2010), that is, very close to the duration of ~ 350 kyr proposed by Friedrich <i>et al.</i> (2003). TOC values are ranging between 1 to 2 wt% and HI values are highly variable (Bréhéret, 1997). Thus, the kerogen-type varies according to each bundle of horizons: Type-II, II-III or III: according to Rock-Eval data, the origin of OM is mixed. At the top of the Fallot Interval, a negative $\delta^{13}\text{C}$ excursion is observed in the Vocontian Basin (Herrle <i>et al.</i> , 2004), which can be correlated with other levels in Tethyan basins (“Livello Renz”, Coccioni <i>et al.</i> , 2006) and in the Atlantic and Pacific Oceans (“Tahlmann” black shales, Herrle <i>et al.</i> , 2003). The Fallot Interval was deposited during a HST (Rubino, 1989; Friès and Parize, 2003). Based on foraminifera, palynomorphs, and kerogen data, two depositional models were proposed by Friedrich <i>et al.</i> (2003). Some of bundles were formed by the “productivity-driven model” (see above) and show Type-II kerogens (marine OM). The other bundles correspond to the “preservation-driven model”, which implies a limited renewal of bottom-water in the basin coupled to an increase of continental runoff and fresh water inputs. Consequently, a water-column stratification developed in the Vocontian Basin, favoring the formation of dysoxic bottom-waters.

Table 1. (continued).

Organic-Rich Levels (OL)	Description
The Jacob Level	<p>The OAE1b was deposited during the Upper Aptian to Lower Albian and is composed of several horizons of black shales in some basins in the Atlantic Ocean and Tethyan Realm (Arthur <i>et al.</i>, 1990; Bralower <i>et al.</i>, 1993, 1999; Erbacher <i>et al.</i>, 2001; Leckie <i>et al.</i>, 2002). In the Vocontian Basin, the Paquier Level was originally considered to correspond to the OAE1b (Bréhéret, 1994, 1997; Herrle <i>et al.</i>, 2003), but, currently, the Jacob, Kilian, Paquier, and Leenhard Levels are now gathered in the OAE1b set (Leckie <i>et al.</i>, 2002; Trabucho Alexandre <i>et al.</i>, 2010; Hu <i>et al.</i>, 2014; Bodin <i>et al.</i>, 2015; Sabatino <i>et al.</i>, 2015, 2018; Ando <i>et al.</i>, 2017). The Jacob Level corresponds to two horizons (35 and 25 cm thick, respectively) of dark marly laminated shales within grey marlstones, over an interval 1 m thick in the Tarendol section (Heimhofer <i>et al.</i>, 2006). On the basis of astronomical calibrations, a duration of ca. 40 kyr was proposed for this OL (Huang <i>et al.</i>, 2010). TOC values are of ~2 wt% on average and Rock-Eval data indicate Type-III kerogens (HI of ~200 mgHC/gTOC; Bréhéret, 1997; Heimhofer <i>et al.</i>, 2006). The strong contribution of terrestrial OM is confirmed by palynofacies observations (Heimhofer <i>et al.</i>, 2006). Contrary to the other OL, neither positive nor negative $\delta^{13}\text{C}$ excursions have been observed for the Jacob Level (Herrle <i>et al.</i>, 2004). The regional equivalent of the Jacob Level is the “Level 113” in the Umbria-Marche Basin (Erba <i>et al.</i>, 1989; Herrle <i>et al.</i>, 2004) and there would exist another equivalent in the Central Atlantic Ocean (Sabatino <i>et al.</i>, 2015). The “preservation-driven model” was proposed for the deposition of the Jacob Level, which is coherent with: 1) the presence of Type-III kerogens (Bréhéret, 1997); 2) palynofacies being dominated by terrestrial particles (Heimhofer <i>et al.</i>, 2006); and, 3) foraminiferal data (Erbacher <i>et al.</i>, 1998). The Jacob Level is interpreted to be a 3rd order MFS (Rubino, 1989; pers. com. in 2017 but see also Friès and Parize, 2003).</p>
The Kilian Level	<p>The Kilian Level is present as a 80 cm-thick horizon of dark marly shales within grey marlstones in the Tarendol section (Bréhéret, 1997). The TOC can reach up to 3 wt% with very low HI values (~100 mgHC/gTOC), corresponding to a terrestrial origin of the OM (Type-III kerogen; Bréhéret, 1994, 1997). According to astronomical calibration, the duration of this thin horizon was estimated to be of ca. 120 kyr (Huang <i>et al.</i>, 2010). In the Vocontian Basin, the Kilian Level corresponds to a strong negative $\delta^{13}\text{C}$ excursion (Herrle <i>et al.</i>, 2004). There are some equivalents of this level, sometimes also called “Kilian Level” in the Umbria-Marche Basin and Central Atlantic (Sabatino <i>et al.</i>, 2015, 2018). The depositional model proposed by Herrle <i>et al.</i> (2003) is very close to the “preservation-driven model”. On account of a monsoon-type forcing, enhanced warm and humid conditions implied increased fresh-water inputs into the Vocontian Basin, and, as a result, the reduction of bottom-water ventilation (Herrle <i>et al.</i>, 2003). The Kilian Level was formed during a TST (Rubino, 1989; pers. com. in 2017).</p>
The Paquier Level	<p>The Paquier Level consists of dark, marly, laminated shales, 1.6 m-thick, within dark marlstones in the L’Arboudeysse section (Herrle <i>et al.</i>, 2003). This OL is observed in numerous locations with an equal thickness (Bréhéret, 1997). TOC values are ranging between 3 to 8 wt%, with HI values up to 500 mgHC/gTOC (Tribovillard, 1989; Bréhéret, 1994, 1997). Type-II kerogens indicate a marine OM, however, palynofacies observation put forward the significant contribution of terrestrial OM (Tribovillard and Gorin, 1991). In the Vocontian Basin, this OL coincides with a strong negative $\delta^{13}\text{C}$ excursion (Herrle <i>et al.</i>, 2004,) and it was estimated to have a ca. 44 kyr duration (Huang <i>et al.</i>, 2010). The Paquier Level corresponds to the “Livello Urbino” in the Umbria-Marche Basin, and has equivalents in the Central and North Atlantic (Coccioni <i>et al.</i>, 2006; Föllmi, 2012). In the Vocontian Basin, the Paquier Level was interpreted as a 3rd order MFS (Rubino, 1989; Friès and Parize, 2003), and, at larger scale, it was contemporaneous of a major transgression in the Tethyan realm (Haq <i>et al.</i>, 1987; Hardenbol <i>et al.</i>, 1998). Moreover, it was deposited during a period of warm and humid conditions with strong fresh-water inputs into the basin. These conditions were similar to those interpreted for the Jacob and Kilian Levels (“preservation-driven model”; OAE1b), but they were more intense and led to anoxic conditions in the deep basin (Tribovillard and Gorin, 1991; Bréhéret, 1997; Erbacher <i>et al.</i>, 2001; Herrle <i>et al.</i>, 2003; Ando <i>et al.</i>, 2017). Owing to strong continental-water inputs that have furthered nutrient inputs, surface productivity increased in the basin.</p>

5.2 Grain-size analysis

Grain-size analysis was performed on 161 samples from the carbonate-free fraction using a Malvern Mastersize 2000[®] laser diffractometer at the Laboratoire d'Océanologie et de Géosciences of the Lille University, following the classical protocol detailed in [Trentesaux *et al.* \(2001\)](#). Precision is about 5% ([Sperazza *et al.*, 2004](#)), but for the finest sizes (< 5 μm), the apparatus is slightly less accurate, according to the manufacturer. The grain-size sorting is the following: cohesive particles, including the clay and the finer silt (< 10 μm); sortable silt or non-cohesive silt (10–63 μm); and sand (63–2000 μm). We considered the fraction below 10 μm as cohesive particles, because of the accuracy of the apparatus for the fine grain sizes (< 5 μm). The analyses were performed on the terrigenous fraction (after acid digestion) and then converted into the bulk sediment composition. Moreover, according to [McCave *et al.* \(1995\)](#), the silt size range (2–63 μm) must be subdivided due to the “mechanical” behavior of particles. Thus, silts thinner than 10 μm have the same behavior as that of clays (cohesive particles) whereas silts coarser than 10 μm (sortable silts) behave as non-cohesive particles. In the present study, the coarser silts are used as current strength indicators ([McCave *et al.*, 1995](#)).

5.3 Clay minerals

In the < 2 μm terrigenous fraction, the clay assemblages have been determined on 155 samples with the standard protocol of the X-Ray Diffraction ([Bout-Roumazielles *et al.*, 1999](#); [Riboulleau *et al.*, 2014](#)), using a Bruker D4 Endeavour apparatus in the Laboratoire d'Océanologie et de Géosciences, Lille University. The relative error margin of the semi-quantitative estimation is ± 5% ([Bout-Roumazielles *et al.*, 1999](#)).

5.4 Rock-Eval

Rock-Eval pyrolysis of 165 samples were performed using a Rock-Eval 6[®] apparatus (see [Behar *et al.*, 2001](#) for details) at the Sorbonne University (ISTeP, Paris). Total organic carbon contents (TOC, wt%), hydrogen index (HI, mgHC/gTOC), oxygen Index (OI, mgCO₂/gTOC) and Tmax (°C) were determined. According to [Espitalié \(1993\)](#), Rock-Eval pyrolysis allows typifying the type and thermal maturity of kerogens. Keeping in mind that the carbonate fraction of the Marnes Bleues Fm. is mainly composed of calcite ([Bréhéret, 1997](#)), total carbonate content (CaCO₃, wt%) has been calculated from the Rock-Eval MinC (%) with the formula: CaCO₃ (%) = 7.976 × MinC (%). To the best of our knowledge, the TOC data available in the literature consulted about the Vocontian Basin were also measured using Rock-Eval pyrolysis.

5.5 Elemental analysis

Determination of the contents in major and trace elements was performed at the University of Lausanne (ISTE-UNIL laboratory) using X-ray fluorescence (XRF) spectrometry, according to the protocol detailed in [Montero-Serrano *et al.*](#)

(2015). Detection limits were < 0.01 wt% for major elements and between 1 and 5 ppm for trace elements. The accuracy of analysis was checked by analysis of standard reference materials.

For the Notre-Dame (*pro parte*), Glaise (*pro parte*), Saint-Jaume, Serre-Chaitieu and Tarendol sections (92 samples), major and traces element analysis was performed at STARLAB Laboratory (Central Michigan University). The elements were analyzed by inductively coupled plasma mass spectrometry (ICP-MS), using external calibration ([Babos *et al.*, 2019](#)). Analytical precision based on replicate analyses was better than 8% and detection limits were < 1 ppb for the major elements and < 40 ppt for the trace elements.

Enrichments factors (EFs) were calculated using the following formula:

$$X_{EF} = (X/Al)_{\text{sample}} / (X/Al)_{\text{upper crust}}$$

where X and Al are the concentrations of element X and aluminum (wt%). Enrichment factors are normalized using the elemental upper crust concentrations of [McLennan \(2001\)](#), and, to minimize the effects of dilution by carbonate or biogenic silica, the aluminum normalization is commonly used (see [Tribovillard *et al.*, 2006](#) for explanations and limits). For example, an EF equal or close to 1 indicates that the sample has a concentration equal to the element concentration in the upper crust. A significant enrichment of an element over average upper crustal concentrations corresponds to EF > 3, while moderate to strong enrichments correspond to an EF > 10 ([Algeo and Tribovillard, 2009](#)).

A chemical weathering index, the Index of Alteration (IA), was calculated. The IA is equivalent to the Chemical Index of Alteration (CIA, [Nesbitt and Young, 1982](#)), and may be used in case of carbonate-rich sediments (> 30% carbonates), as the CIA may lead to misleading interpretations ([Goldberg and Humayun, 2010](#)). IA = LN (Al₂O₃/Na₂O), in molar proportions. This index avoids uncertainties concerning the necessary corrections caused by the presence of non-silicate phases, in particular, the carbonate and phosphate phases ([Von Eynatten *et al.*, 2003](#)). Increased IA values can be interpreted as reflecting more intense chemical weathering over emerged lands, source areas of the clastic fraction.

5.6 Average Sedimentation Rate (SR) and Total Organic Carbon Mass Accumulation Rates (TOC MARs)

Previous works calculated an average sedimentation rate (SR) in the Upper Aptian of about 3 cm/kyr, regardless of the compaction rates ([Köbler *et al.*, 2001](#); [Heimhofer *et al.*, 2004](#)). This average SR was used for most of the samples corresponding to the background hemipelagites in the present study. For the organic-rich levels, the average SR were specifically calculated with the following formula: average SR (cm/kyr) = thickness/duration of the organic-rich levels, where thickness was measured in cm ([Bréhéret, 1997](#); [Dauphin, 2002](#); [Herrle *et al.*, 2003](#); [Heimhofer *et al.*, 2006](#); [Stein *et al.*, 2011](#); [Westermann *et al.*, 2013](#)) and duration was estimated from orbital calibrations in kyr ([Köbler *et al.*, 2001](#); [Huang *et al.*, 2010](#); [Malinverno *et al.*, 2010](#); see [Table S3](#)). For the

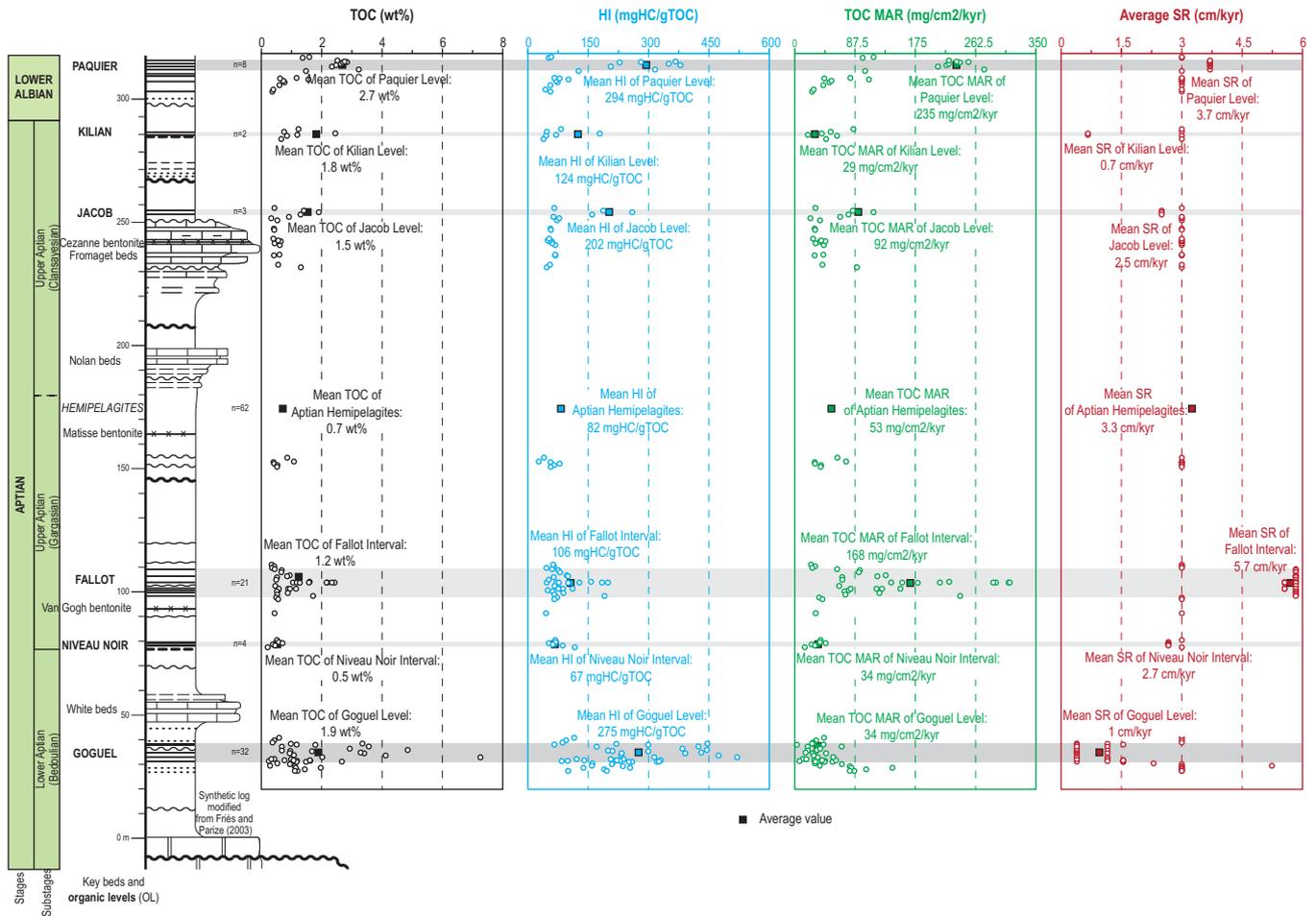


Fig. 4. TOC, HI, SRs and TOC MARs: (A) within the sequence-stratigraphy framework; and, (B) in the Aptian to Lower Albian interval. *Fig. 4. TOC, HI, SR et TOC MAR: (A) dans le cadre de la stratigraphie séquentielle ; et (B) dans l'intervalle de l'Aptien à l'Albien inférieur.*

Note-Dame section, where the top of the Goguel Level is not observed (Friès and Parize, 2003) and, for the Baudinard section, for which no calibration data are available, sedimentation rates could not be calculated.

To evaluate the variations of organic carbon inputs, the TOC mass accumulation rate (TOC MAR) can be calculated using the following formula from Westermann *et al.* (2013):

$$\text{TOC MAR (mg/cm}^2\text{/kyr)} = (\text{TOC (wt\%)} \times \text{rock density (g/cm}^3\text{)} \times \text{average SR (cm/kyr)}) \times 10$$
, where TOC values come from Rock-Eval data, the rock density is 2.3 g/cm³ for marlstones, 2.4 g/cm³ for siltstones and 2.7 g/cm³ for marly limestones and limestones (Attewell and Farmer, 1976; marlstones if CaCO₃ > 35%, marly limestones and limestones if CaCO₃ > 65% in this study).

Organic matter dilution by clastic materials in the dark levels was calculated according to data available in previous works (Tribovillard and Gorin, 1991; Bréhéret, 1997; Heimhofer *et al.*, 2004; Ando *et al.*, 2017). The TOC, thickness, and duration of each dark level are available in Table S4. It is important to precise that the aim of calculating these accumulation rates (average SR and TOC MAR) is to inter-compare the sections. Direct comparison with accumulation rates of modern environments or other geological

formations is not recommended because compaction rates are not considered here in the average SR calculation.

6 Results

The results are presented in Tables S3 and S5–S9. In order to facilitate data screening, the main results associated to the average values within the dark levels and background hemipelagites, are shown in Figures 5 and 6. Box-plot diagrams are used to compare the samples according to their positions in the sequence stratigraphy framework (Fig. 3).

6.1 Sediment characteristics

The mode of the grain-size distribution curves (clastic fraction of the rock) ranges from 8 to 85 μm (Table S5). The dark levels exhibit an average mode slightly lower (20 to 26 μm) than that of the hemipelagites (32 μm), except for the Falloit Interval (42 μm; Figs. 3 and 4). Sediments are mainly composed of siltstones (carbonate content < 35%) and marlstones (carbonate content > 35%; Table S5). The distribution of particle sizes follows a unimodal normal

distribution. The proportions of cohesive particles are lower in the hemipelagites with Falloit Interval and Paquier Level displaying lower values (19, 18, and 17% on average, respectively) than the other dark levels (23 to 26%, Figs. 3 and 4). The proportions of sortable silts are similar or slightly lower for the hemipelagites (38%), compared to the dark levels (39 to 44%). The average proportions of sands are lower than 10% in both hemipelagites and dark levels, except for the Falloit Interval (14%; Figs. 3 and 4). Carbonate contents show significant variations (21 to 35%), with a gradual increase of the CaCO₃ proportion (also previously mentioned by Br  h  ret, 1997).

The comparison of the system tracts and MFS displays slightly higher average modes in the LST and HST (38 and 35 μm , respectively) than in the TST and MFS (29 and 24 μm , respectively). Similarly, the samples corresponding to TST and MFS show weakly higher proportions of cohesive particles (21 and 25% on average) than the LST samples (17%, Figs. 3 and 4), while the average proportion of cohesive particles in the HST is 20%. The LST exhibits the lowest average proportions of sortable silts (34%), coupled to the highest proportions of carbonates (41%). On the contrary, the average proportions of sortable silts are dominant in the TST, MFS, and HST (40 to 43%). The MFS and HST exhibit slightly lower carbonate content (24 and 28%) than the TST (31%).

6.2 Clay minerals

The distribution of clay minerals is dominated by illite-smectite mixed-layer minerals (I-S R1, with smectite layers amounting to less than 40%), illite, and smectite (I-S R0 with smectite layers making up to 70–90% of the edifice), in decreasing proportions (Table S5). The clay-mineral assemblages also display subordinate proportions of chlorite and kaolinite, and the ratio of these two minerals fluctuates according to orbital parameters (Ghirardi *et al.*, 2014). Let us remind that kaolinite is generally a good indicator of variations in the hydrological cycle, an important factor for understanding the deposition of OM-rich levels (*e.g.*, Corentin *et al.*, 2020). The Goguel, Kilian, Niveau Noir and Falloit levels and hemipelagites show high average contents of I-S, ranging from 32 to 42%. In these groups of samples, average smectite contents are fluctuating from 10% in the Niveau Noir Interval to 30% in the hemipelagites, while the Goguel Level is devoid of smectite in the Sauzeries section (Table S6; this is expected, the section being in the deeply buried part of the basin; see Caillaud *et al.*, 2020). The clay assemblages of the Jacob and Paquier levels are dominated by smectite (44 and 42% on average, respectively), with significant I-S contents (24 and 28%). The average illite proportions range from 14%, in the Paquier Level, to 31%, in the Niveau Noir Interval, with a value of 23% in the hemipelagites. Kaolinite and chlorite are present in minor abundance (< 15% on average), except for the Kilian Level (kaolinite content of 18% on average). Through the Aptian to Lower Albian interval, two main variations are observed in the clay assemblage: (1) smectite gradually increases (although the Kilian Level exhibits a local decrease in smectite contents, the caging sediments show high contents); and (2) I-S and illite gradually decrease upward. To a lesser extent, the kaolinite content weakly increases.

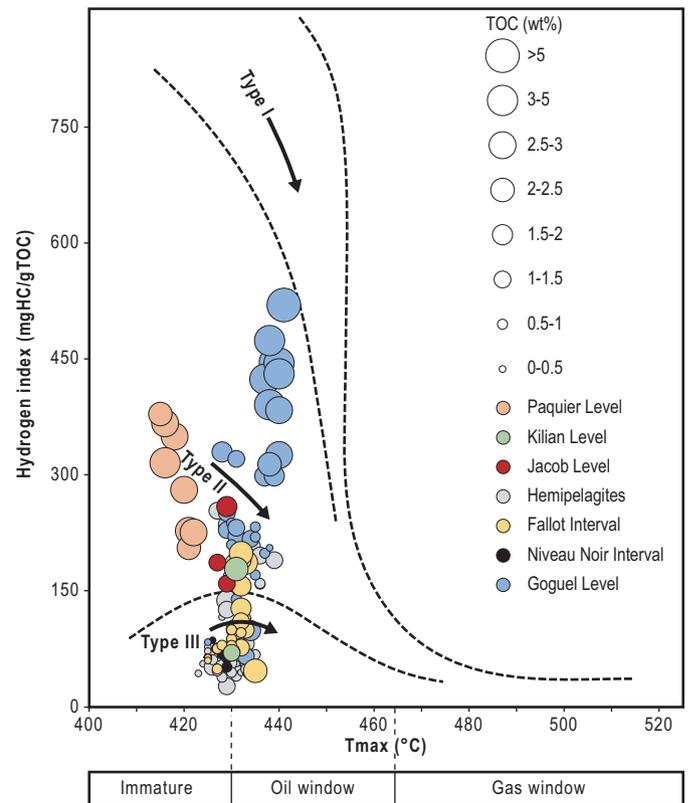


Fig. 5. Pseudo Van-Krevelen diagram (Espitali   *et al.*, 1985) showing the typology of the OM for the studied samples.

Fig. 5. Pseudo diagramme de Van-Krevelen (Espitali   *et al.*, 1985) montrant la typologie de la MO pour les   chantillons   tudi  s.

Box-plot diagrams show some specificity in the distribution of the main clay minerals (Figs. 3 and 4). The smectite contents are higher in the LST and TST (26 and 36% on average, respectively) than in the MFS and HST (20 and 22%). The I-S contents progressively increase from the LST to the HST (30 to 39%). The TST shows slightly lower illite proportions (19%) than the other groups (24 to 26%).

6.3 Bulk organic matter

The total organic carbon (TOC) content displays values ranging from 0.2 to 2.7 wt% (Tables S2 and S7). The hemipelagites show low TOC values (0.2 to 2.0 wt%; 0.7 wt% on average), while the average TOC value of the dark levels (Fig. 5) indicates poor to moderate OM enrichments, ranging from 0.5 wt% in the Niveau Noir Interval to 2.7 wt% in the Paquier Level (see Table S2 for average TOCs and extreme values). The highest HI values are observed in the Goguel (275 mgHC/gTOC on average) and Paquier levels (294 mgHC/gTOC), and the other groups of samples indicate low to very low average HI values (67 to 202 mgHC/gTOC). Tmax values are variable according to the position of sections in the Vocontian Basin, ranging from 415°C to 441°C. Because almost all the samples (131/132) display a TOC > 0.3 wt%, the HI and Tmax parameters could be interpreted in a pseudo Van-

Krevelen diagram (Fig. 5, Espitalié *et al.*, 1985). Thus, the Tmax values indicate immature to oil-window stages for the kerogens. The various types of kerogens suggest various origins of the OM in the Marnes Bleues Fm. corresponding to marine OM (Type-II kerogens), mixed marine or terrestrial OM (Type-II-III kerogens), and terrestrial or degraded marine-OM (Type-III kerogens) (see details for each group of samples in Table S2). Here, the LST and HST show (very) low average TOC (0.6 and 0.9 wt%, respectively) and HI values (55 and 88 mgHC/gTOC), while the TST displays low average TOC (1 wt%) and HI (116 mgHC/gTOC). The samples of MFS show the highest average TOC and HI values (1.9 wt% and 256 mgHC/gTOC).

6.4 Inorganic geochemistry

Elemental concentrations and enrichment factors (EFs) are reported in Tables S8 and S9. For the Marnes Bleues Fm., the Fe/Al ratio is ranging from 0.29 to 1.16, but the average ratios are approximately similar, for each dark level (0.42 to 0.45), except for the Kilian Level (0.35). These average ratios are close to the average crustal value (0.44, McLennan, 2001). The Ti/Al ratios were calculated to detect a possible excess of titanium compared to aluminum, another useful detrital elemental proxy (Tribovillard *et al.*, 2006). The Ti/Al ratios range from 0.04 to 0.07 (Table S9), which is quite close to the average crustal value. The productivity-sensitive elements (Ba, Cu, Ni) show a homogeneous distribution throughout the Aptian to Lower Albian interval. Average Ba EFs, Cu EFs and Ni EFs display ranges from 0.6 to 1.6, 1.5 to 4, and 1.5 to 2.5, respectively. The only significant (yet low) enrichment factors (EF > 3) are observed for the average Cu EFs in the Kilian Level (3) and in the Niveau Noir Interval (4).

The values of the index of alteration (IA) gradually increase through the stratigraphic interval studied (Fig. 4). In the lower part of the type section, most of the samples show an IA of ~3.6, while in the upper part, the samples display an IA of ~3.8, which is a significant (but modest) variation, regarding this index.

The redox-sensitive elements do not show important variations, except for molybdenum (Fig. 4). The highest Mo EF values were recorded in the Paquier and Jacob levels (22 and 9 on average, respectively), and, to a lesser extent, the Goguel Level (2 on average, but 5 on average in the Sauzeries section, which is not much). In the other groups of samples, the average Mo EFs range from 0.4 to 2.3 (that is, no significant enrichment). Uranium displays a weak enrichment in the Paquier Level (EF=3.8 on average), but it exhibits almost invariant EFs in the other groups of samples (0.8 to 1.7). The average EFs of Co, Cr, and V, do not show any significant enrichment, neither in the dark levels nor in the hemipelagites (0.7–0.9, 1–1.3, 1.2–2.1).

6.5 Average Sedimentation Rate (SR) and Total Organic Carbon Accumulation Rates (TOC MARs)

The average SRs values range from 0.4 to 5.8 cm/kyr. The hemipelagites show an average SR value of 3.3 cm/kyr, and only the Paquier Level and the Fallot Interval exhibit a higher average SR (Table S2; Fig. 6), while the Jacob Level and the

Niveau Noir Interval display a slightly lower average SR than the hemipelagites. The Kilian and the Goguel levels yield low average SRs.

The TOC MARs show variable values (3 to 312 mg/cm²/kyr, average values in Table S3): (1) low average TOC MARs in the Goguel Level, Niveau Noir Interval, hemipelagites and Kilian Level (Table S2); (2) moderate average TOC MAR in the Jacob Level (Table S2); and, (3) high average TOC MARs in the Fallot Interval and Paquier Level (Table S2). The LST and TST exhibit moderate average SRs (2.8–3 cm/kyr), whereas the HST shows the highest average SR (4.8 cm/kyr). The MFS samples correspond to low average SR (1.8 cm/kyr). The mean TOC MARs increase progressively from low values in the LST and moderate values in the TST-MFS to high values in the HST (Table S2).

7 Discussion

Prior to this study, most of the works dealing with dark levels within the Vocontian Basin (Weissert, 1990; Tribovillard and Gorin, 1991; Bréhéret, 1994; Föllmi, 1995; Sanfourche and Baudin, 2001; Friedrich *et al.*, 2003; Herrle *et al.*, 2003, 2010; Heimhofer *et al.*, 2006; Stein *et al.*, 2011; Westermann *et al.*, 2013) were largely inspired by the usual two depositional models: the “productivity-driven model” and the “preservation-driven model” (Fig. 7; Schlanger and Jenkys, 1976; Demaison and Moore, 1980; Bralower and Thierstein, 1984; Arthur *et al.*, 1990; Pedersen and Calvert, 1990; Erba, 1994; Erbacher *et al.*, 1996, 1998, 2001; Premoli Silva *et al.*, 1999; Leckie *et al.*, 2002; Jenkyns, 2010; among others). Obviously, these traditional models partly explain the depositional conditions of the dark levels in the Marnes Bleues Fm., but recent works (Sageman *et al.*, 2003; Harris, 2005; Bohacs *et al.*, 2005; Burdige, 2005, 2006, 2007; Caillaud *et al.*, 2020 and references therein) demonstrated that other factors must be considered such as: (1) the nature of OM (marine vs. terrestrial) (2) the dilution or concentration of OM in sediments (sedimentation rate, turbidites), (3) the geographical context of the Tethys Ocean (limited bottom-water renewal and possible inflow of trace-metal- and oxygen-depleted waters) and (4) the tectono-sedimentary context. The latter point is essentially concerned with relative sea-level variations that may be disconnected from eustatic variations because of all the twists and turns occurring during sedimentary basin evolution. Therefore, the depositional model may be updated in the light of the present results.

Before considering the various dark levels individually, some broad features may be derived from our results.

7.1 Impact of burial

From the Rock-Eval data, the OM maturity of the Marnes Bleues Fm. is low (immature stage to onset of oil window), except for the Baudinard section (upper end of oil window). The higher OM maturity of the Baudinard section may be explained by a thick burial and the proximity of the Alpine Thrust Front, as formerly deduced from the clay assemblages in the eastern part of the Vocontian Basin (Levert and Ferry, 1987, 1988; Deconinck, 1984, 1987; Levert and Ferry, 1988; Ghirardi *et al.*, 2014; Caillaud *et al.*, 2020). Based on

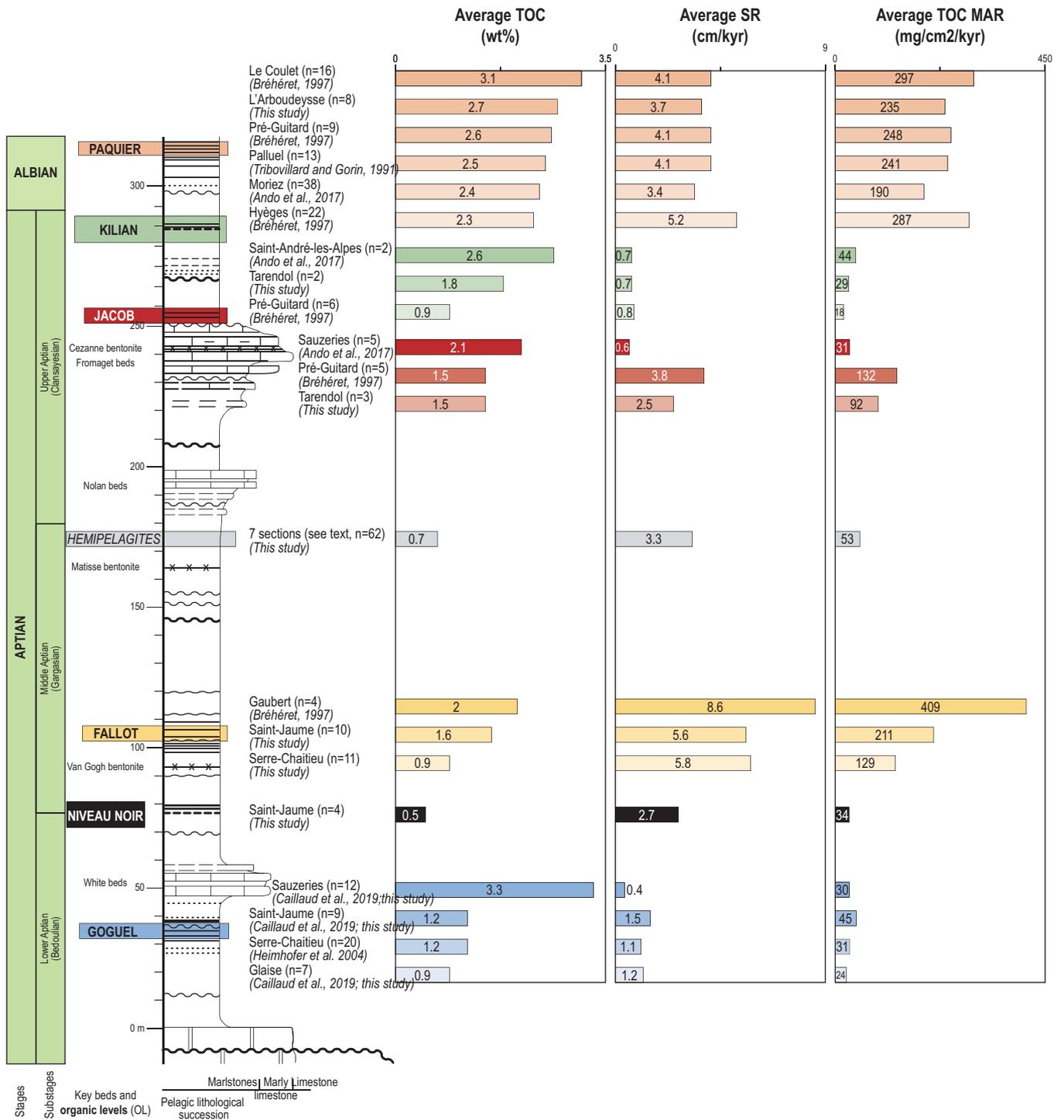


Fig. 6. Comparison of average TOC, SR, TOC MARs between the dark levels of the Aptian to Lower Albian interval, data from several works (Tribovillard and Gorin, 1991; Bréhéret, 1997; Heimhofer *et al.*, 2004; Ando *et al.*, 2017; Caillaud *et al.*, 2020).

Fig. 6. Comparaison des MAR COT, SR, COT moyens entre les niveaux riches en MO de l'intervalle Aptien-Albien inférieur; données issues de plusieurs travaux (Tribovillard et Gorin, 1991; Bréhéret, 1997; Heimhofer *et al.*, 2004; Ando *et al.*, 2017; Caillaud *et al.*, 2020).

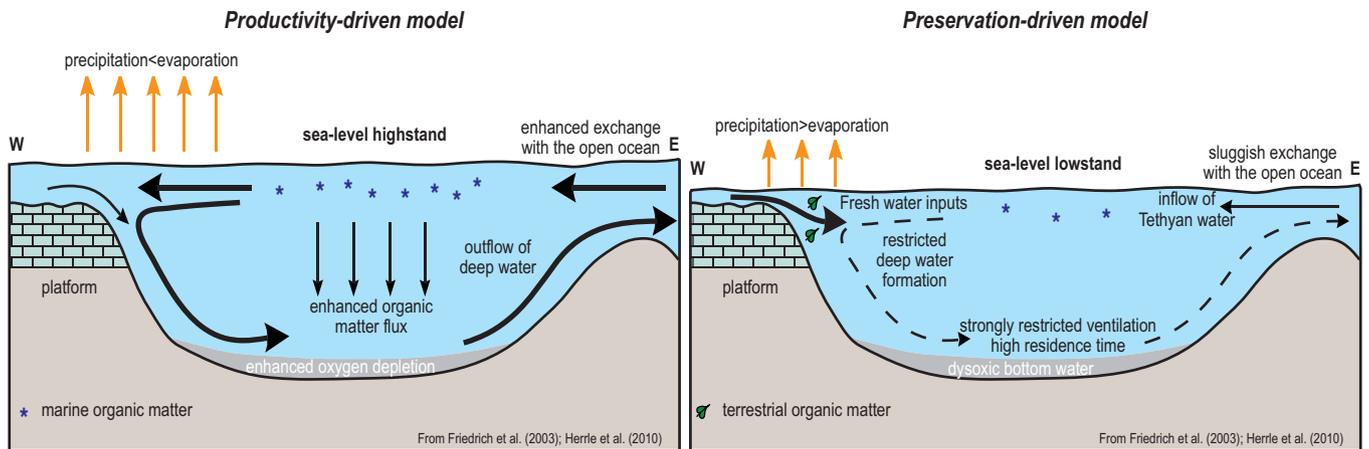


Fig. 7. Depositional model of dark levels of the Marnes Bleues Fm. (modified from Friedrich *et al.*, 2003; Herrle *et al.*, 2010).
Fig. 7. Modèle de dépôt des niveaux riches en MO des Marnes Bleues (modifié de Friedrich *et al.*, 2003; Herrle *et al.*, 2010).

clay-assembly examination, Levert and Ferry (1987) estimated the burial depth in the western part of the Vocontian Basin to range between 500 and 1000 m. Basing on unpublished data (Caillaud's PhD thesis) and on-going studies (Quijada *et al.*, to be published), the low thermal maturity of the OM in the Marnes Bleues Fm. is also deduced from the biomarker isomerization ratios of the saturated fractions, as well as aromatic compounds such as phenanthrene and methylphenanthrene isomerization ratios (allowing vitrinite reflectance to be calculated at $\sim 0.63\%$), which correspond to the beginning of the oil window..

7.2 Conditions of oxygenation

Largely, the redox-sensitive elements such as U, Mo, V, or even Cr show enrichment factors keeping to low values (below or close to the threshold limit) with some exceptions discussed below. After Algeo and Tribouillard (2009), it means that most of the samples point to oxic to occasionally suboxic conditions of deposition. The basin bottom did not undergo durably anoxic conditions and never reached euxinic conditions. In addition, a U-EF vs. Mo-EF diagram (Fig. 8; Algeo and Tribouillard, 2009) shows that some samples witnessed paleoenvironmental conditions prone to the particulate shuttle process. In a few words, in such situations, Mo may be conveyed to the sediments through capture onto iron and/or manganese oxy-hydroxides settling through a water column containing dissolved O_2 . Molybdenum was kept trapped within the sediments because reducing conditions developed at the sediment-water interface or at shallow distance below it (Fig. 8).

7.3 Nature of OM, productivity and climate-induced nutrient supply

Rock-Eval parameters show highly variable HI values in the Marnes Bleues Fm. (27 to 707 mgHC/gTOC) corresponding to Type-II, Type-II-III and Type-III kerogens. These variable parameters suggest multiple origins for OM. The molecular study (Quijada *et al.*, to be published elsewhere) shows that all the levels studied here contain a mixture of

marine OM and terrestrial OM, present in various proportions. The Paquier, Kilian and Fallot levels are the most enriched in land-derived OM.

In the Marnes Bleues Fm., the absence of authigenic enrichments of productivity-sensitive elements (Ni-, Cu-, Ba-enrichment factors close to 1 and almost-always < 3) indicates that surface-water productivity was low to normal during the deposition of the formation. Nevertheless, the proportion of smectite, within the clay-sized fraction of the sediment, and the IA, calculated from the bulk detrital fraction of the sediments, increased through the Aptian to Lower Albian interval. These variations could indicate enhanced nutrient inputs into the basin, through the increase of: (1) clay mineral abundance formed under moderately to strongly hydrolyzing on-land conditions (smectite and kaolinite; Chamley, 1989; Basilone *et al.*, 2017); and, (2) IA values, related to chemical hydrolysis on land (Von Eynatten *et al.*, 2003). Despite these observations, the increased IA and smectite proportion do not correlate with any enrichment of productivity-sensitive elements. These results suggest a climatic change evolving toward more hydrolyzing conditions from the Aptian to the Lower Albian.

7.4 Sequence stratigraphy

Organic-matter sedimentation may be placed in the sequence stratigraphy framework of the Marnes Bleues Fm. Thanks to the numerous samples studied here (LST, 20 samples; TST, 26 samples; MFS, 47 samples and HST, 39 samples), the sequence-stratigraphy interpretation can be somewhat refined. The sequence-stratigraphy pattern was designed from sedimentological considerations; here we can add some independent arguments. The interpretation of the distribution of clay minerals and IA is questionable because these factors display a long-term vertical trend throughout the Aptian to Lower Albian interval. The sediment composition fits well with the published sequence-stratigraphy framework: 1) the lowest average grain-size mode corresponds to the MFS (24 μm), whereas the highest modes are in the LST and HST (38 and 35 μm , respectively); 2) the highest proportions of cohesive particles and sortable silts are located in the MFS (25

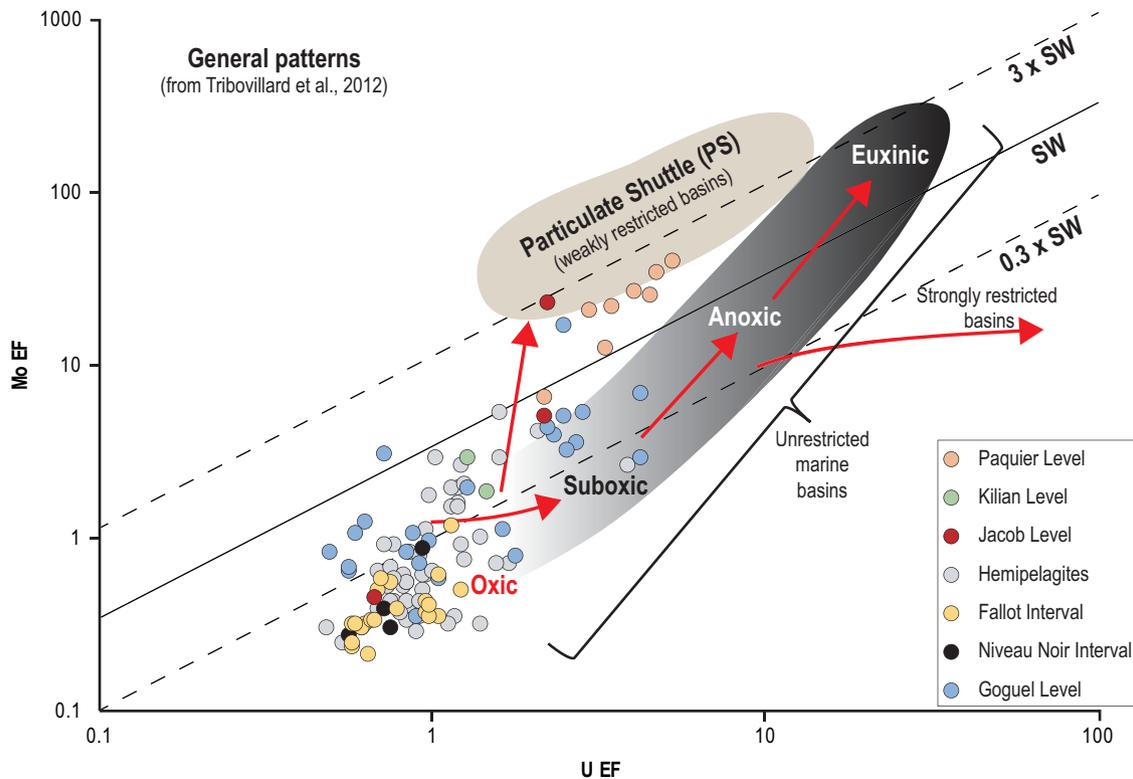


Fig. 8. Plot of U-EF vs. Mo-EF for the GL samples showing the oxygenation conditions in the Aptian to Lower Albian of the Marnes Bleues Fm. The SW lines corresponds to the Mo/U molar ratios similar to the seawater value (SW \sim 7.5–7.9) and the dashed lines correspond to the fractions thereof (3xSW, 0.3xSW). The pattern of U and Mo EFs are compared to the model proposed in [Algeo and Tribovillard \(2009\)](#).

Fig. 8. Tracé de U-EF vs. Mo-EF pour les échantillons GL montrant les conditions d'oxygénation dans l'Aptien à l'Albien inférieur des Marnes Bleues Fm. Les lignes SW correspondent aux rapports molaires Mo/U similaires à la valeur de l'eau de mer (SW \sim 7,5–7,9) et les lignes en pointillés correspondent à leurs multiple ou fraction (3xSW, 0,3xSW). Le modèle des FE U et Mo est comparé au modèle proposé dans [Algeo et Tribovillard \(2009\)](#).

and 43%, respectively); and, 3) the highest average carbonate contents correspond to the LST (41%) because the production of carbonates was restricted to the basin during the LST ([Rubino, 1989](#); [Friès and Parize, 2003](#)). Several trends are also observed in the OM sedimentation. First, the highest TOC (1.9 wt% on average) and HI (256 mgHC/gTOC on average) are recorded during the MFS: most of the dark levels correspond to MFS. In the same way, the redox-sensitive molybdenum displays some enrichment in the MFS, which denotes low-oxygen deep waters during the MFS. The productivity-sensitive elements show a moderate enrichment of Cu in the LST, probably related to the high biogenic-carbonate contents. The SRs were also consistent with the system tract, with the highest average SRs in the LST and HST (3 and 4.8 cm/kyr) and the lower average SR in the MFS (1.8 cm/kyr). Nevertheless, the highest TOC MARs do not occur in the MFS (76 mg/cm²/kyr) but in the HST (114 mg/cm²/kyr). Thus, the tectono-sedimentary-controlled variations of the sea level are accurately recorded by the organic content of the dark levels, as reported by [Basilone \(2020, 2021a\)](#) for similar contexts of deposition. Therefore, it is assumed that the MFS display the best conditions for OM preservation (low oxygenation and SRs) in the Marnes Bleues Fm. Another evidence is that all dark levels were deposited in a high relative

sea-level context (TST, MFS and HST), implying that a low relative sea level (LST) was not favorable to OM preservation.

7.5 The dark levels considered individually

The dark level can be assessed each in turn, after an initial look at the background sedimentation. The main factors identified for each level are summarized in [Figure 9](#).

The *hemipelagites* correspond to the background sedimentation of the Marnes Bleues Fm. Their OM content is low (mean TOC = 0.7 wt%) and they were deposited under oxic conditions with low to normal productivity. Based on molecular biomarker analysis, the *hemipelagites* present mostly algal-bacterial OM, and Type-III to Type-II/III kerogens, implying a notable contribution of continental OM and/or degraded marine-OM. The *hemipelagites* show slightly higher grain-size modes than most of the dark levels and they correspond to marlstones while the dark levels correspond to siltstones. The average SR and TOC MARs of *hemipelagites* are used as a baseline to be compared with other SRs and TOC MARs in the Marnes Bleues Fm.

The *Goguel Level* shows contrasting results depending on the sections examined. These results were intensively discussed in [Caillaud *et al.* \(2020\)](#), therefore only a summary

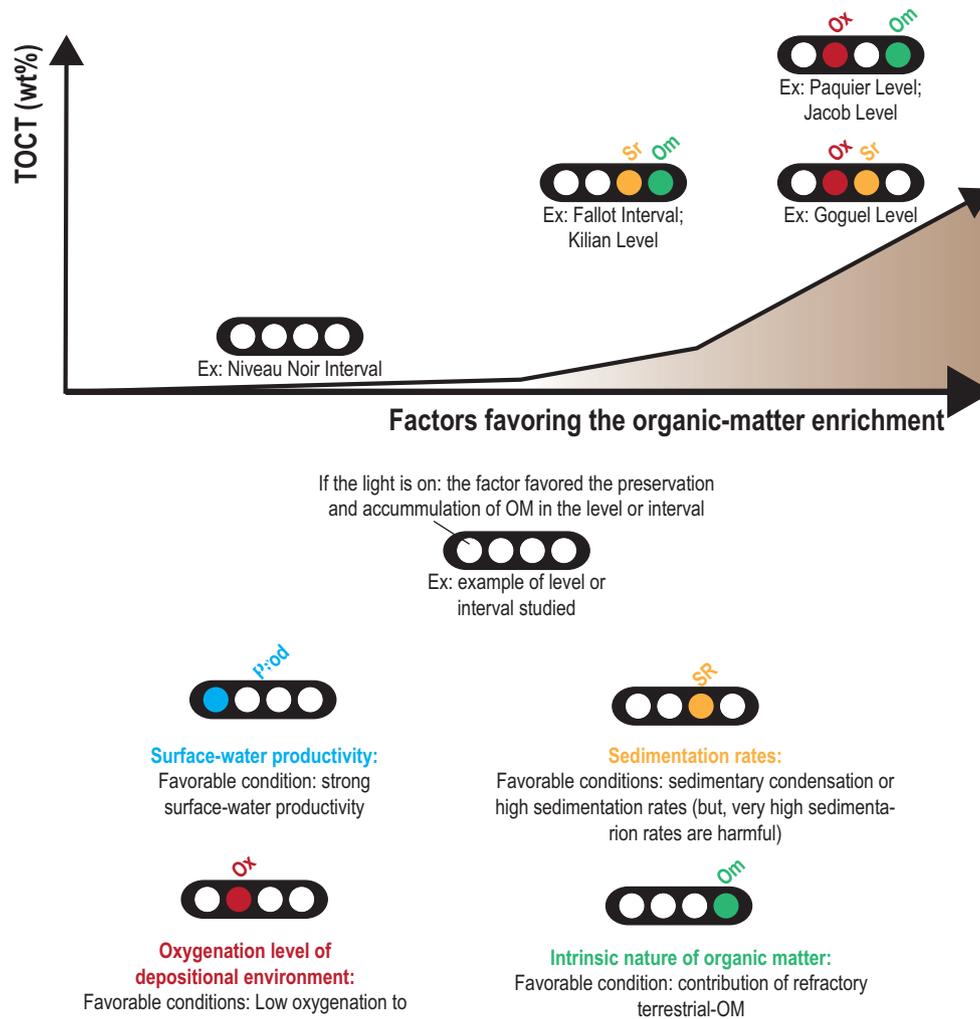


Fig. 9. Synthesis of factors and processes that controlled OM sedimentation in the Aptian to Lower Albian interval of the Marnes Bleues Fm. *Fig. 9. Synthèse des facteurs et processus contrôlant la sédimentation de la MO dans l'intervalle Aptien à Albien inférieur des Marnes Bleues.*

of the main interpretations is presented here. First, although the average TOC is 1.9 wt%, the section of Sauzeries exhibits high average TOC (3.3 wt%) and HI (387 mgHC/gTOC) but a low average TOC MAR (30 mg/cm²/kyr). Second, the surface-water productivity was low to normal, but the oxygenation conditions were heterogeneous. As demonstrated by the redox-sensitive elements and molecular biomarkers, in the proximal environments the conditions were oxic, while in the distal settings the conditions were lowly oxygenated. In addition, the biomarker data suggest the water column was intermittently stratified with hypoxia/anoxia occasionally developing into the photic zone. In the same way, although the OM was mainly of marine origin, a significant terrestrial contribution was evidenced in the proximal environments through biomarker examination. Third, the Goguel Level was deposited during a period of sedimentary condensation indicated by the low average SRs (0.4 cm/kyr to 1.5 cm/kyr) compared to the hemipelagites. In more detail, the very low average SR at Sauzeries (0.4 cm/kyr) corresponds to the section devoid of turbidites, on the contrary of the Notre-Dame, Saint-Jaume, Glaise and Serre-Chaitieu sections, where thin turbidites are

interbedded. In addition to the sedimentary condensation, the low average grain-size mode and the high proportions of cohesive particles and sortable silts (27 and 44% on average, respectively), compared to the hemipelagites, are consistent with the 3rd order MFS position in a sequence stratigraphy framework (Rubino, 1989; pers. com. 2017; Bréhéret, 1997; Ferry, 2017).

To summarize, the depositional model of the Goguel Level in the Vocontian Basin encompasses three distinct environments (Caillaud *et al.*, 2020): (1) a proximal area (Saint-Jaume and Notre-Dame sections) where mixed-origin OM was diluted by turbidites and remineralized (oxic conditions); (2) a distal area (Glaise section) where marine OM was diluted and remineralized (both process induced by turbidites) under low-oxygen conditions; and, (3) a distal area (Sauzeries section) without turbidites, where marine OM was condensed and preserved under low-oxygen conditions. Therefore, in the case of the Goguel Level, although the mixed nature of OM contributed to a slightly more efficient preservation in the proximal areas, the two factors that significantly contributed to OM enrichment are sedimentary “condensation” and low

“oxygenation level” (stratified basin with oxygen-poor bottom-waters).

The *Niveau Noir Interval* was categorized as an organic-rich level in the literature, but it is not enriched in OM (aver. TOC of 0.5 wt%; aver. HI of 67 mgHC/gTOC). Organic matter is mostly composed of marine OM without significant contribution from terrestrially-derived biomarkers, deposited under oxic conditions with modest SRs (2.7 cm/kyr on average). Although [Herrle *et al.* \(2010\)](#) proposed a period of strong surface-water productivity coupled to evaporation), our new dataset indicates that surface-water productivity was normal to moderate, because only Cu exhibits a detectable (yet rather weak) enrichment factor. The Niveau Noir Interval is thus definitely not an organic-rich level, because “no factor” significantly contributed to OM enrichment. The results of grain-size analysis display an average mode of 20 μm and sediments composed of relatively abundant cohesive particles and sortable silts (26 and 42% on average, respectively) compared to the hemipelagites. Therefore, this fine-grained sediment composition is consistent with the sequence-stratigraphy framework, where the Niveau Noir Interval is interpreted as a 3rd order MFS ([Rubino, 1989](#); [Friès and Parize, 2003](#)).

The *Fallot Interval* shows low to moderate average TOC (1.2 wt%, 0.6 and 1.6 wt% in the studied sections) with relatively low average HI (106 mgHC/gTOC). Rock-Eval data indicate the presence of Type-III kerogens. Molecular biomarkers indicate an important contribution of terrestrially-derived OM. The inorganic geochemistry and biomarkers clearly indicate that the Fallot Interval was deposited under oxic conditions with low to normal surface-water productivity. However, the average SR (5.7 cm/kyr) and TOC MARs (168 mg/cm²/kyr) were very high compared to hemipelagites. In the same way, the grain-size mode indicates relatively high values (42 μm on average), as is the case for the average sand proportion in the sediments (14%). The high SRs and the coarser grain size of the Fallot Interval are consistent with the HST position proposed by [Rubino \(1989\)](#) and [Friès and Parize \(2003\)](#). The depositional model proposed for the Fallot Interval (alternation of the “preservation-driven model” with the “productivity-driven model”, [Friedrich *et al.*, 2003](#)) is not consistent with our new findings. Consequently, we propose the following factors influenced OM enrichment: (1) dilution (because of the very high SRs); and, (2) oxygenation conditions (*i.e.*, oxic in the case of the Fallot Interval). According to [Tyson \(2001, 2005\)](#), in oxic environments, increased SRs can be correlated with increased organic-carbon burial. The oxygen level is considered only a minor process in this case because, under low-oxygen conditions, high SRs are harmful to the organic contents in that they act as a diluting agent ([Tyson, 2001, 2005](#)). Nevertheless, even if the SRs of the Fallot Interval were high, they did not fall in the range proposed by [Tyson \(2005\)](#), *i.e.*, more than 20 cm/kyr. Therefore, OM preservation was probably enhanced by one additional factor: the “nature of OM”. Organic matter was partly composed of land-derived, refractory organic compounds. This model is coherent with the data of [Bréhéret \(1997\)](#) regarding the Gaubert section: the highest average SRs match with the highest TOCs. To summarize, the moderate OM enrichment for the Fallot Interval is the consequence of the fast burial of terrestrial OM under oxic conditions.

The *Jacob Level* shows moderate organic contents (mean TOC of 1.5 wt%) of Type-II to-III kerogen. This level contains mixed OM with an important contribution of land-derived products, inferred from molecular data. In addition, palynofacies data indicate high contributions of terrestrial OM ([Heimhofer *et al.*, 2006](#)). In the studied section (Tarendol), the average SR (2.5 cm/kyr) and the surface-water productivity were normal compared to the background sedimentation (hemipelagites). In the Pré-Guittard section (data from [Bréhéret, 1997](#)), approximately the same values of average SR, TOC MAR, and TOC were reported. However, inorganic geochemistry indicates that bottom conditions were poorly oxygenated (at least periodically) during the Jacob Level. The biomarkers corroborate this assumption. The mode of the grain-size distribution (21 μm , that is, lower than for hemipelagites) and the proportions of cohesive particles and sortable silts (23 and 39%, higher than for hemipelagites) are coherent with the position within the sequence-stratigraphy framework (MFS). The depositional model proposed by [Heimhofer *et al.* \(2006\)](#), that is, the “preservation-driven model”, is consistent with the evidences of low-oxygenated conditions demonstrated here. Nevertheless, we propose that the refractory nature of the terrestrial OM deposited favored OM preservation. To conclude, the enrichment of OM in the Jacob Level resulted from two major parameters: the “oxygenation level” (low-oxygenated bottom-waters) and the “nature of OM” (refractory continental OM). In this dark level, the addition of a minor factor, the low dilution (favoring sedimentary condensation of OM), led to the highest organic contents (mean TOC of 2.1 wt%) in the Sauzeries section. This point, plus the nature of the OM, illustrates the role played by the relative sea-level variations at this regional scale.

The *Kilian Level* contains Type-III kerogens (124 mgHC/gTOC) and the organic contents show a moderate enrichment of 1.8 wt% on average. In the sequence-stratigraphy framework, this dark level is interpreted as a HST, which is in adequacy with the slightly lower mode values (26 μm on average) and the higher proportions of cohesive particles and sortable silts (23 and 43% on average, respectively) compared to the background sedimentation. The OM from this interval is composed of marine products, with a significant presence of terrestrial biomarkers deriving from conifers [Quijada *et al.*, ongoing study](#)). Our new results suggest the Kilian Level was deposited under oxic conditions. However, the work of [Okano *et al.* \(2008\)](#) found tri- and penta-methylcosanes (TMI and PMI) in the Kilian Level at the Sauzeries section (same samples as those studied by [Ando *et al.*, 2017](#)). Such TMI and PMI indicate the occurrence of methanogenic archaea and, therefore, low-oxygen conditions at the sediment/water interface. Surface-water productivity was low to normal. In the Tarendol section, the low average SR (0.7 cm/kyr) is interpreted as a sedimentary condensation, and, according to [Bréhéret \(1997\)](#) and [Ando *et al.* \(2017\)](#), the Sauzeries and Pré-Guittard sections yield average SRs of the same range of values. Hitherto the depositional model assigned to the Kilian Level was the “preservation-driven model” ([Herrle *et al.*, 2003](#)), but our results are not totally concordant with this model. We propose that the main two parameters that contributed to OM preservation were the “dilution level” (here: condensation of OM) coupled to the “nature of OM” (here: contribution of refractory terrestrial OM). The

depositional model proposed by Herrle *et al.* (2003) could be consistent locally for the Sauzeries section, where the highest average TOC values are recorded (2.6 wt%, using data of Ando *et al.*, 2017). In addition to sedimentary condensation, the likely low-oxygen conditions favored the preservation of terrestrial and marine OM in the Kilian Level.

The Paquier Level shows uniformly “high” average TOC (2.3 to 3.1 wt%) and moderate SRs (3.4 to 5.2 cm/kyr). The Van-Krevelen-type diagram indicates Type II-III to Type-II kerogens for the samples studied in the L'Arboudeysse section. Biomarkers suggest marine OM with the highest relative contribution of terrestrial OM including leaf waxes. The contribution of terrestrial OM was also demonstrated with palynofacies data (Tribovillard and Gorin, 1991). Biomarker results clearly show that the Paquier Level was deposited under intermittent anoxic conditions. In addition, our results meet those of Benamara *et al.* (2020) when they point to conditions favoring the particulate shuttle-induced transfer of Mo to the sediments, which indicates depositional setting conditions with an oxygenated water column and the developments of reducing conditions at shallow depth below the sediment-water interface. Based on the interpretation of the productivity-sensitive elements, surface-water productivity was low to normal. At first, this interpretation seems to be inconsistent with the high average TOC MARs calculated (190 to 297 mg/cm²/kyr), however, we assume that moderate SRs coupled to a significant proportion of refractory continental OM deposited under low-oxygen conditions allowed for this range of values. The sediment composition of the Paquier Level is partially consistent with a 3rd order MFS in the Vocontian Basin. Indeed, even if the average mode of the grain-size is lower in the Paquier Level (26 μm) than in the hemipelagites, sediment composition is relatively similar to that of hemipelagites. Our new insights partially support the previous depositional model proposed by Herrle *et al.* (2003) with a significant productivity and a strong continental runoff (Tribovillard and Gorin, 1991; Benamara *et al.*, 2020), as no increase of surface-water productivity is observed. The deposition of OM in the Paquier Level was governed by two main parameters: the “oxygenation level” (recurrent anoxic conditions) and the “nature of OM” (here mixed origin of OM). For the latter factor, it is conceivable that the labile marine OM was preserved during the anoxic period in addition to land-derived OM, while the refractory terrestrial OM alone was preserved during the suboxic-oxic periods. In addition, a high accumulation of OM (TOC MARs) was the outcome of moderate SRs associated with high continental runoff. The wholesale constancy of SRs (based on 6 sections) is consistent with previous works of Bréhéret (1997), demonstrating that the Paquier Level has a large geographic extension throughout the Vocontian Basin.

8 Regional and global controls

The weathering proxies (smectite and IA) show a progressive increase during the entire deposition of the Marnes Bleues Fm. Either for the IA or smectite, this trend is interpreted as an intensification of chemical weathering affecting the clastic-sediment sources on land. This long-term evolution was probably induced by climate warming, in particular during the latest Aptian. It is consistent with the

nannofossil-based temperatures variation curve reconstructed by Godet *et al.* (2008), Bottini *et al.* (2015) and Bottini and Erba (2018) for the western Tethys, and also with the TEXH86-SST data of O'Brien *et al.* (2017). In addition, it is proposed here that one process could have been superimposed onto the clay mineral record and terrigenous fraction, namely, the Vocontian Basin long-term evolution. According to previous works (Bréhéret, 1997; Ferry, 1999, 2017; Friès and Parize, 2003), the Vocontian Basin was progressively constricted during the Aptian and Albian by the uplift of the Vivarais and Dauphiné platforms and the uplift of the Durancian Isthmus. Consequently, the source-to-sink distance decreased during the Aptian to Lower Albian. Thus, we propose that: (1) the Goguel Level recorded a distal signature of mixed terrigenous-materials, because of the larger distance from sedimentary sources, also reinforced by a global high sea level during the OAE1a (Haq *et al.*, 1987; Hardenbol *et al.*, 1998; Haq, 2014); (2) the progressive closure of the Vocontian Basin allowed the climate changes on the closer-and-closer sediment sources to be recorded. To summarize, the nature of OM, and more particularly the relative contribution of continental OM in the dark levels, was controlled by: (1) the climatic conditions that could have stimulated the production of terrestrial OM (warm and humid conditions); and (2) the tectonic settings, which must have favored the export of terrestrial OM, depending on the distance to the coasts, evolving through time. The omnipresent control by the sea-level variations driven by the tectonic-sedimentation interaction must not be forgotten, as reminded at various places above.

In the same way, marine primary productivity could have been controlled by the conjunction of climatic conditions and local basin physiography. In the Marnes Bleues Fm., according to our results, surface-water productivity was low to normal. Incidentally, Gambacorta *et al.* (2020) have the same conclusion for an equivalent of OAE1d, namely, the Pialli Level of the Umbria-Marche Basin (central Italy). This basin also accumulated dark grey to black shales under oxic to suboxic conditions. Therefore, like for the nature of OM, surface-water productivity may have resulted from: (1) specific climatic conditions favoring nutrient export; and, (2) specific basin physiography favoring nutrient concentration.

Unlike the dilution and nature of OM, the oxygenation level of the Vocontian Basin may have been controlled by global processes in addition to regional factors. The oxygenation factor seemed closely linked to the second order eustatism and to the submarine-volcanic activity: (1) the Goguel Level resulted from a global high sea level during the emplacement of the Ontong-Java Plateau (OAE1a); (2) the Jacob Level was formed during the maximum of the global regression in the Tethys Ocean coupled to the Kerguelen Plateau; (3) the Paquier Level was deposited after a return to a global high sea-level context, contemporaneous of the Nauru and Kerguelen Plateaus, as illustrated by the works of Bottini *et al.* (2015) and Bottini and Erba (2018). Thus, the periods of deposition of the Goguel, Jacob, and Paquier levels were marked by brief hyperthermal events, associated with the large igneous provinces (LIP). These global events may have triggered low-oxygen conditions during Mid-Cretaceous interval (Bodin *et al.*, 2015; Vickers *et al.*, 2016).

The global high sea level (Paquier and Goguel levels) could have favored the development of oxygen-depleted deep areas of the semi-enclosed Vocontian Basin. In the case of the global sea-level fall in the Tethys Ocean (Jacob Level), the Vocontian Basin could have been partly isolated, involving a limited renewal of deep waters (as already mentioned by Br  h  ret, 1994). Concurrent regional or global factors must have occurred, each of them yielding contrasting periodicities or durations, which entangled the sedimentary record. Thus global, LIP-related (thermal) events may have conditioned the oxygen-level of oceanic water masses and sea-level variations may have conditioned the renewal of the Vocontian water masses, depending on the type of exchanges between the Tethys Ocean and the relatively isolated Vocontian Basin.

From numerous works related to Tethyan basins (*e.g.*, Coccioni *et al.*, 1990, 2006; Baudin *et al.*, 1998; Leckie *et al.*, 2002; F  llmi, 2012; Sabatino *et al.*, 2015, 2018) and the North Atlantic (Trabucho Alexandre, 2011), the comparison of organic-rich levels in the Marnes Bleues Fm. (Goguel, Jacob, Kilian and Paquier levels; TOC of 1–3 wt% on average) and their time equivalents (Livello Selli, Level 113, Kilian Level and Livello Urbino; TOC > 3 wt% on average) reveals that: (1) the nature of OM was totally different from that of the Marne Bleues background, being exclusively marine, confirming that the nature of OM was partly controlled by local environmental factors and paleogeography; (2) SRs were logically lower in the deep oceans compared to the Vocontian Basin (0.22 to 0.47 cm/kyr for the Level 113 to Livello Urbino, Sabatino *et al.*, 2018; and 0.12 to 0.27 cm/kyr for the Selli Level, Westermann *et al.*, 2013), supporting that dilution depended on the location within the Tethys Ocean (platform *vs.* slope *vs.* deep basin); (3) the depositional conditions were dysoxic to anoxic in all black-shales time-equivalents, induced by the emplacement of large igneous provinces, (4) surface-water productivity was strong in all black-shale being time-equivalents of the dark levels of the Vocontian Basin (even if it was variable in the Kilian Level between the North Atlantic and the Tethys Ocean, (Trabucho Alexandre *et al.*, 2011; Sabatino *et al.*, 2015), highlighting a global control like for the oxygenation conditions (probably the volcanic activity too, Larson and Erba, 1999; Leckie *et al.*, 2002; Trabucho Alexandre *et al.*, 2010, 2011). We can thus hypothesize that the black shales of the Tethys Ocean were richer in OM because they were more condensed and the productivity was more intense than in the Vocontian Basin.

9 Conclusion

Because the Vocontian Basin was a medium-size basin accumulating thick and apparently homogenous successions of marly/shaly sediments, the OM-rich levels studied here could have been expected to share the same controls. In addition, the semi-enclosed disposition of the basin is supposed to have favored the development of oxygen-limited conditions. Surprisingly, our study demonstrates that each dark level resulted from a different scenario and that the organic content was low (TOC < 3%). The various parameters influencing OM storage differ from one level to one another: proportions of marine *vs.* terrestrial OM, sedimentation and accumulation rates, redox conditions, productivity, sea level, etc. This intra-basinal fluctuation of the factors impacting OM accumulation

is to be considered in a larger-scale framework, where regional to global factors also evolve: tectonics modifying the basin geometry, climate, (eustatic) sea-level variations, LIP emplacements, etc. Nevertheless, all dark levels of the Vocontian Basin were deposited under low-to-normal conditions of productivity and all levels (but the Paquier Level) underwent oxic to suboxic conditions. The factors favoring the accumulation of OM other than productivity and anoxia have been brought into play, either in turn or in synergy, in the various situations illustrated by the organic levels of the Vocontian Basin. Nevertheless, it is observed that in the absence of productivity and/or anoxia favorable to preservation, these other factors only account for (very) low enrichments in OM. They would therefore only be capable to modulate the response of the organic record but not to cause marked enrichments. Therefore, a semi-enclosed environment such as that of the Vocontian Basin did not favor the development of oxygen-restricted conditions with a modest productivity, although additional factors enhancing OM accumulation were operating. Thus our results strongly suggest that hemipelagic environments collecting thick successions of marly/shaly sediments cannot accumulate OM in significant proportions if a high productivity is absent from surface waters.

These results discussed here are not typically specific to the Cretaceous Vocontian basin that displays quite common features, *i.e.*, a medium-sized, semi-enclosed basin where hemipelagic sediments accumulated below a water depth of a few hundreds of meters. Consequently, we argue that our conclusion could be applied to similar basins, independently of their time of formation and location.

Supplementary Material

Table S1. Details for log-sections sampled (coordinates and sources). Localization in.

Table S2. Synthesis of OM, SRs and TOC MARs data. For details, see Table S7.

Table S3. Accumulation rates calculated for the sections studied from literatures and positions of samples in the sequence-stratigraphy framework.

Table S4. Accumulation rates calculated from literatures.

Table S5. Grain-size data (in % of the terrigenous fraction and in % of the bulk sediment).

Table S6. Clay mineral composition (in % of the clay-mineral fraction and in % of the bulk sediment).

Table S7. Rock-Eval parameters and the carbonate contents calculated from the MINC (%).

Table S8. Geochemical data (major and trace elements) of the studied samples. < LD means below limit of detection.

Table S9. Geochemical data (Al-normalized concentrations and enrichment factors) of the studied samples. < LD means below limit of detection. EF stands for enrichment factor (see text).

The Supplementary Material is available at <http://www.bsgf.fr/10.1051/bsgf/2022001/olm>.

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