Enhanced chemical weathering and organic carbon burial as recovery factors for the OAE2 environmental conditions: a case study from Koppeh-Dagh Basin, NE Iran

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Abstract
A late Cenomanian-early Turonian interval (Hamam-ghaleh section) adjusted with the transition of Aitamir and Abderaz formations has been investigated in the east of Koppeh-Dagh Basin to examine geochemical anomalies and environmental perturbations related to the oceanic anoxic event 2 (OAE2). The study succession is composed of dark gray shale and glauconitic sandstone of Aitamir Formation, which is conformably overlain by cream marl and marly limestones of Abderaz Formation. The dark shale of the upper Aitamir Formation indicate higher organic matter concentrations especially in the two intervals at the end of *Rotalipora cushmani* biozone and at the middle part of *Whiteinella archaeocretacea* biozone. These intervals are characterized by higher detrital input (quartz and feldspar) and chemical alteration (high kaolinite/illite ratios), which suggest a warm-humid condition coeval with high productivity during the OAE2. The Aitamir Formation also shows higher total sulfur (TS) values associated with deposition of (quartz and feldspar) and chemical alteration (high kaolinite/illite ratios), which suggest a warm-humid condition coeval with high productivity during the OAE2. The Aitamir Formation also shows higher total sulfur (TS) values associated with deposition of... 

Keywords: Cenomanian/Turonian Boundary, Chemical Weathering, Koppeh-Dagh, OAE2, Organic Carbon Burial.

Introduction
Global changes in climate, environment and oceanography leave the signatures in geochemical proxies of marine or terrestrial sedimentary records (Weissett et al., 2008). Characterization of depositional units and their correlation based on stratigraphic and geochemical variations initiated usage of the term “chemostratigraphy” that have been more frequent from the 1980s (Ramkumar, 2015). The mid-cretaceous represents one of the most extensive geochemical anomalies in the Phanerozoic Eon, which has been largely studied based on chemostratigraphic analysis (Schlanger and Jenkyns, 1976; Arthur et al., 1985; Leckie et al., 2002; Jarvis et al., 2006; Voigt et al., 2006; Jenkyns, 2010; Jarvis et al., 2011, 2015 among others). During this episode dysoxia and anoxia referring to Oceanic Anoxic Events (OAEs; Schlanger and Jenkyns, 1976) occurred episodically across the marine basins (e.g., Schlanger and Jenkyns, 1976; Arthur et al., 1985; Takashima et al., 2009; Jenkyns, 2010). OAE2 at the Cenomanian/Turonian boundary (CTB; 93.6 Ma, Ogg et al., 2008) is one of the most prominent OAEs, which is characterized by the widespread organic rich sediments (Takashima et al., 2009) (Fig. 1-A), worldwide documented positive carbon isotope excursion in both carbonate (δ13C) and organic matter (δ13Corg) (e.g., Scholle and Arthur, 1980; Arthur et al., 1988; Jarvis et al., 2006, 2011; Voigt et al., 2006; Jenkyns, 2010; Gavrilov et al., 2013), negative excursion in nitrogen isotope ratio (δ15N) (e.g., Junium and Arthur, 2007; Meyers et al., 2009; Baroni et al., 2015), and concentration of redox sensitive elements (e. g., Turgeon and Brumsak, 2006; Gavrilov et al., 2013).

The CTB in the Koppeh-Dagh Basin have spread out between Aitamir and Abderaz formations. This boundary have been studied for biostratigraphy and palaeoecology of planktonic and benthic foraminifera (Abdoshahi et al., 2010; Ghoorchaei et al., 2011; Kalanat et al., 2016, 2017b, 2018b) and palaeoenvironmental changes based on nitrogen isotope variations (Kalanat et al., 2017a). The main objective of our study is to evaluate the environmental perturbation across OAE2 on the Hamam-ghaleh section in the east of Koppeh-Dagh...
Organic-carbon isotope ($\delta^{13}$C$_{org}$), total organic carbon (TOC) and total sulfur contents (TS) are combined with mineralogy data to provide an integrated chemostratigraphic correlation between the study section and other CTB successions in the koppeh-Dagh Basin and present a regional model to describe palaeoenvironmental condition in the basin.

Geological setting and study area
Koppeh-Dagh Basin
The formation and evolution of numerous continental blocks of Iran and surrounding area have been mainly controlled by the extensive orogenic activities during the opening and closure of Palaeotethys and Neotethys oceans (Bagheri and Stampfili, 2008). Palaeotethys was the ocean that separated Eurasian plate from the Gondwana (Muttoni et al., 2009). The northward subduction of this ocean generated the slab-pull forces on the Gondwana plate that led to detachment of Cimmerian blocks (including Iran, central Afghanistan, Karakoram, Pamir, etc.) and opening of Neotethys ocean (Bagheri and Stampfili, 2008). Continuing subduction of Palaeotethys ocean and northward drifting of the Cimmerian blocks finally led to collision of Iranian plate and Turan plate (Eurasia) around the middle-late Triassic resulting in the early Cimmerian orogeny (Bagheri and Stampfili, 2008; Robert et al., 2014). After this collision the deposition of Koppeh-Dagh Basin started on the southern margin of Eurasian plate in a narrow shelf and continental slope environments. This basin covers an area of over 500 km$^2$ in the northeast of Iran (Fig. 1B) with more than 7000 m-thick sediments including carbonate, siliciclastic and evaporate from Jurassic to Neogene (Afshar-Harb, 1979; Robert et al., 2014).

The Aitamir (Albian-Cenomanian) and Abderaz (Turonian to early Campanian) are two widespread upper Cretaceous formations in the Koppeh-Dagh Basin (Fig. 2). There is a conformable transition between the two formations based on planktonic foraminiferal studies and lithostratigraphic data in the east of Koppeh-Dagh Basin (Mokhtari et al., 1999; Abdoshahi et al., 2010; Goorchaei et al., 2011; Kalanat et al., 2016).

Hamam-ghaleh section
The Hamam-ghaleh section is located in the east of Koppeh-Dagh Basin, 7km south to Kalate-Nader City, near the Hamam-ghaleh village (Fig. 1B and 1C). The studied succession is composed of 26 m-thick dark gray shale and glauconitic silty sandstone of Aitamir Formation with low abundance of benthic and planktonic foraminifera and high concentration of frambooidal pyrite. It is conformably overlain by 22 m-thick cream marl and marly limestones of Abderaz Formation, which are characterized by high diversity of foraminifera and lack of frambooidal pyrite.

The section has been previously studied based on planktonic foraminifera (Goorchaei et al., 2011), and three biozones, namely, Roralipora cushmani, Whiteinella archaeocretacea and Helvetoglobotruncana helvetica (Premoli-Silva and Verga, 2004) have been reported, spanning from late Cenomanian to early Turonian. The CTB lies in the W. archaeocretacea biozone, but the exact horizon of the boundary cannot be specified by planktonic foraminifera.

Materials and Methods
36 powdered and de-carbonated samples (8 shale, 4 sandstone, 25 marl and marly limestone) were analyzed for TOC, TS and $\delta^{13}$C$_{org}$ at Department of Earth and Planetary Science, University of Tokyo (Table 1). For C and S analysis, ca. 20 mg of each samples were measured using a Thermo Finnigan Flash EA 1112 series CNS analyzer using a retention time of 720 s. The analytical error was less than 0.2 wt% for TOC, and <0.1 wt% for TS, using sulfamethazine standard.

Ca. 1.5 mg of samples were analyzed for $\delta^{13}$C$_{org}$ by a combination system of Thermo Finnigan Flash EA 1112 series CNS analyser using a retention time of 720 s. The analytical error was less than 0.2 wt% for TOC, and <0.1 wt% for TS, using sulfamethazine standard.

Bulk rock mineralogy was determined using an Ultima IV X-Ray Diffractometer at Department of Earth and Planetary Science, University of Tokyo.

Results
Total organic carbon and total sulfur (TOC and TS)
TOC values in the Hamam-ghaleh section range between 0.083% and 0.83%. Two organic-rich intervals occur at the end of R. cushmani and the middle part of W. archaeocretacea biozones. Generally, the Aitamir Formation shows relatively-higher organic matter contents with the average value of 0.5%, whereas TOC values in the Abderaz Formation are low and oscillate around a mean of 0.13%. The Aitamir Formation also shows mean
sulfur content of 0.07% and two outliers of 0.32% and 0.24% occur at samples HM5 and HM10 respectively, while the TS contents in the most samples of the Abderaz Formation are almost 0% (Table 1, Fig. 3).

Figure 1. a- Distribution of black shales and organic-rich sediments around the Cenomanian/Turonian boundary (Modified after Takashima et al., 2009). The position of Hamam-ghaleh section is specified by H. b- Map of Iran and surrounding areas. Neotethys and Paleotethys sutures zones, Koppeh-Dagh Basin and study area are indicated (modified after Angiolini et al., 2007). c- Locality map of Hamam-ghaleh section in 7 km south of Kalate-Nader city.

Figure 2. A part of 1/100000 geological map of Kalate-Nader in the east of Koppeh-Dagh Basin (Nabavieh, 1998). The location of Hamam-ghaleh section laying in the Aitamir-Abderaz formations transition is indicated by star.
Table 1. TOC, TS and δ¹³Corg of the late Cenomanian-early Turonian sequence in Hamam-ghaleh section, Koppeh-Dagh Basin.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Height (m)</th>
<th>TOC (%)</th>
<th>TS (%)</th>
<th>δ¹³Corg (‰)</th>
<th>Sample No.</th>
<th>Height (m)</th>
<th>TOC (%)</th>
<th>TS (%)</th>
<th>δ¹³Corg (‰)</th>
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<td>0.45</td>
<td>0</td>
<td>-</td>
<td>HM18</td>
<td>30</td>
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<td>0</td>
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<tr>
<td>HM1</td>
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<td>0.73</td>
<td>7.6×10⁻³</td>
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<td>HM19</td>
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<td>-26.67</td>
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<tr>
<td>HM2</td>
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<td>0.07</td>
<td>-27.02</td>
<td>HM20</td>
<td>31</td>
<td>0.19</td>
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<tr>
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<td>-</td>
<td>-</td>
<td>HM36</td>
<td>48</td>
<td>0.15</td>
<td>0</td>
<td>-27.09</td>
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Figure 3. Geochemical data from the Hamam-ghaleh section including total organic carbon (TOC), total sulfur (TS), carbon isotope in organic matter fraction (δ¹³Corg), kaolinite/illite ratio as chemical weathering proxy and bulk rock mineralogy.
Mineralogy
The bulk rock compositions in the lower part of the studied section (Aitamir Formation) consist mostly of quartz (61-78%) and feldspar (plagioclase + Kfeldspar, 15-24%). Minor components include calcite (0.7-11%) and clay minerals (3-10%). A marked increase in calcite (50-80%) and decrease in detrital components (quartz and feldspar) have been observed in the upper part of the section (Abderaz Formation). This change reflects the lithological transition from shale and sandstone of Aitamir Formation to marlstone and marly limestone of Abderaz Formation (Fig. 3).

Carbon isotopes
The $\delta^{13}$C$_{org}$ values in the studied section varies between $-25.85\%$ and $-27.35\%$. The values show a positive excursion to $-26.52\%$ at the end of $R$. cushmani biozone (HM4), and then decrease down to $-27.35\%$ at the middle part of $W$. archaeocretacea biozone (HM9), followed by an increase to $-26.21\%$ at sample HM13. After that the $\delta^{13}$C$_{org}$ curve shows a negative trend until the top of the studied succession (Tab.1, Fig.3).

Discussion
TOC and TS in the Hamam-ghaleh section
Generally organic matter burial is mainly controlled by primary productivity and preservation. Organic carbon flux to the sea-floor is dependent on such variable factors as enhanced runoff, intensified upwelling and fertilization of oceanic water by volcanically derived metals like Fe. On the other hand, preservation proposes reduced decomposition of organic matter as a result of decreased bottom water oxygen levels and/or high sedimentation rate (Demaison and Moore, 1980; Canfield, 1994; Meyers, 1997; Meyers et al., 2006). In reality, both productivity and preservation have a crucial role in forming black shale deposits, but the plankton productivity is commonly the most important (Pedersen and Calvert, 1990, Jenkyns et al., 2002).

OAE2 is characterized by widespread deposition of black shale in the deep water, upwelling zones (e.g. Voigt et al., 2006; Junium & Arthur, 2007; Mort et al., 2008) and restricted areas (e.g. Leckie et al., 1998; Elderbak et al., 2014), however some shallow water and coastal areas are black shale-free (Keller et al., 2008; Gertsch et al., 2010). CTB interval is also characterized by high TS contents (Wortmann et al., 1999; Brumsack, 2006; Okano et al., 2007; Hetzel et al., 2009), because the oxygen deficiency increases sulfate reduction and sulfide formation like pyrite in the sediments (Dean et al., 2013).

The Hamam-ghaleh section contains two TOC-enriched intervals (Fig. 3). The lower and larger one (up to 0.8%) is at the base of section (end of $R$. cushmani biozone) and the second one covers the upper part of Aitamir Formation at the middle part of $W$. archaeocretacea biozone. The TOC-enriched intervals accompanied by higher TS contents and presence of frambooidal pyrite, implying the effect of OAE2 and expansion of oxygen deficiency in the upper Aitamir Formation, whereas the total sulfur in the following interval (Abderaz Formation) decline to zero percent (except for the sample HM32) (Fig 3). Beside the expansion of oxygen minimum zone, higher TOC contents in the Aitamir Formation could be result of higher accumulation rate and/or delivery of the terrestrial organic matter into the basin.

Comparison of TOC profile in the Hamam-ghaleh section with two other sections in the east of Koppeh-Dagh basin (Gharesu and Taherabad sections; Fig. 4) reveals that black shale is not deposited in this part of basin. The TOC contents and position of organic-enriched intervals in the Aitamir Formation are almost similar (Kalanat et al., 2017b; Kalanat et al., 2018b).

Mineralogy and palaeo-climate condition
Kaolinite and illite are two major clay mineral components in the Hamam-ghaleh section. Kaolinite as a product of chemical weathering forms under humid condition (Partrish, 1998; Schnyder et al., 2005; Gertsch et al., 2010), whereas illite is an indicative of moderate climate and low intensity of chemical alteration (Singer, 1984; Oliveira et al., 2002). The concept of kaolinite/illite ratio, which is called “clay mineral index” can be used as palaeo-climate proxy (Robert and Kennet, 1994, 1997; Mahmudy Gharnie et al., 2004) in chemostratigraphy.

Kaolinite/illite ratio in the study succession indicates relatively higher values in the Aitamir Formation especially in the two organic matter-enriched intervals. Higher clay mineral index accompanied with large values of detrital minerals in the Aitamir Formation reflect a warm and humid condition with higher chemical weathering and enhanced runoff into the basin in the late Cenomanian. A dramatic decrease in detrital input (deposition of carbonate dominant sediments) from Aitamir up to the Abderaz formations, and
relatively lower kaolinite/illite ratios can be interpreted as a cooler and drier climate with reduced chemical weathering and runoff into the basin.

**Carbon stratigraphy of the CTB**

Carbon isotopes are not so sensitive to diagenetic alterations, therefore potentially provide excellent information about changes in global carbon cycle, particularly variation in the burial rate of organic matter. It also can be used as palaeoenvironmental proxy for volcanogenic CO₂ supply, weathering rate, changes in ocean circulation and release of methane from the clathrate in the sea floor (Pearce et al., 2009).

The CTB interval is characterized by a major global positive excursion of δ¹³C that occurs in marine carbonate, and both marine and terrestrial organic matter (Arthur et al., 1988; Jarvis et al., 2006, 2011; Caron et al., 2006). This is mainly attributed to the widespread removal of isotopically light carbon (¹²C) from the carbon cycle, due to increased biological production and/or increased preservation of organic matter (Schlanger and Jenkyns, 1976; Scholle and Arthur, 1980; Arthur et al., 1985, 1988). The δ¹³Corg values are also partially controlled by the isotope fractionation associated with photosynthesis (ɛ_p), whereas organic matter provided in lower atmospheric pCO₂ is relatively ¹²C-depleted (van Bentum et al., 2012).

In the CTB, superimposed on the long-term positive carbon excursion, some major features in δ¹³C pattern can be identified. Such general pattern in δ¹³C data has been first reported by Pratt and Threlkeld (1984) as an initial enrichment (“A”), a brief recovery (“B”), and a sustained plateau (“C”), which subsequently found in a globally correlation (e.g., Jarvis et al., 2006). The alternative view is that δ¹³C profile across the CTB includes three peaks, where the upper two peaks appear as two small projections on a broad peak (e.g., Caron et al., 2006; Takashima et al., 2009; Pearce et al., 2009; Jarvis et al., 2011).

During the past decades, the carbon stratigraphic correlation has been examined using biostratigraphic datum levels (e.g., Bengtson, 1996; Caron, 2006; Jervis, 2011). These studies indicates that the last δ¹³C peak occurs around the CTB, but minor differences are attributed to preservation factors associated with facies changes and/or difficulties in precisely placing the datum levels (Caron et al., 2006).

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**Figure 4. TOC and δ¹³Corg in the Hamam-ghaleh, Gharesu and Taherabad sections. Horizontal lines show correlation of major carbon isotope peaks. The geochemical data for Gharesu section are from Kalanat et al. (2018b) and for Taherabad section are based on Kalanat et al. (2018a). The green band indicates interval of higher TOC bearing deposits and positive δ¹³C excursion defining Oceanic Anoxic Event 2.**
At the Hamam-ghaleh section the excursion begins at the top of R. cushmani biozone (sample HM4), followed by a brief recovery around the middle part of W. archaeocretacea biozone (HM7-HM12), and a longer positive excursion at the top of this biozone (HM13-HM18). The excursion ends at the upper part of section in the H. helvetica biozone (Fig 3). The second and the last large peak in the study succession occurs after sandstone unit and is considering as position of CTB.

Comparison of $\delta^{13}C_{\text{org}}$ values to other sections
the CTB $\delta^{13}C_{\text{org}}$ excursion shows the amplitude as much as 4‰ in many localities such as Atlantic region (Arthur et al., 1988; Kuypers et al., 2002; Sinninghe Damsté et al., 2008) Boreal Sea (Tsikos et al., 2004) and Tethys Ocean (Jarvis et al., 2011). The $\delta^{13}C_{\text{org}}$ excursion value in the study area is about 1‰, whereas the other CTB Koppeh-Dagh sections in the Gharesu and Taherabad (Kalanat et al., 2018a, b), also reveal greater positive shifts about 2.5‰ and 2‰, respectively (Fig. 4). These differences may be caused by varying sedimentation rate of terrestrial organic matter in the shallow environments, which were transported from continent into the basin.

Apart from the larger $\delta^{13}C_{\text{org}}$ positive shifts in the Gharesu and Taherabad sections than the study succession, the sharp excursion and stratigraphic position of major shifts in the upper part of R. cushmani and W. archaeocretacea biozones are comparable. The recovery phase after the first excursion seems too negative in these sections (even more negative than post OAE2 interval) in compare to other sections in the world (e.g. Caron, 2006; Jervis, 2011), which can be explained by enhanced fluvial delivery of isotopically light inorganic carbon recycled from soil organic matter. Only some of these material would need to be oxidized and make the isotopic composition of photic zone carbon pool lighter (Meyers et al., 2006). This hypothesis can be confirmed in the study area by correspondence of the intervals characterizing by enhanced run off and low $\delta^{13}C_{\text{org}}$ values (Fig. 6).

**Palaeoenvironmental model**
Figure 5 is a simple diagram representing the factors controlling palaeoenvironmental perturbation and climate changes during the OAE2. It proposes that the event was triggered by emplacement of Caribbean Plateau providing CO2 out-gassing and consequence greenhouse condition (Arthur et al., 1985; Larson, 1991; Leckie et al., 2002; Snow and Duncan, 2005; Seton et al., 2009), acceleration of hydrological cycle and enhanced chemical weathering (Fletcher et al., 2008; Barclay et al., 2010; Pogge von Strandmann et al., 2013), high nutrient input, biological productivity and carbon flux to the sea floor (Jones and Jenkyns, 2001), which finally led to fostering the organic matter-enriched sediments.

The high burial rate of carbon in the organic-enriched deposits of CTB was a powerful factor to draw down atmospheric CO2 and cause the consequent cooling of the global climate (Arthur et al., 1988; Freeman and Hayes, 1992; Kuypers et al., 1999). Weathering of silicate minerals also can remove CO2 from the atmosphere (For example, $2\text{CO}_2 + 4\text{H}_2\text{O} + \text{CaAl}_2\text{Si}_2\text{O}_8 \rightarrow \text{Ca}^{++} + 2\text{Al} (\text{OH})_3 + 2\text{SiO}_2^{aq} + 2\text{HCO}_3^{-}$; Brady, 1991) and reduce the temperature (Pogge von Strandmann et al., 2013). Therefore, it can be concluded that the warm and humid intervals with high burial of organic carbon and intensified chemical weathering during the CTB could subsequently turn into cold and drier intervals (Fig. 5).

This process can be followed in our study succession (Fig. 6). Two high TOC intervals coincide with warm-humid conditions, high chemical weathering and high detrital input. We propose that surface runoff provide low-density fresh water cap, which led to water column stratification. These conditions coevol with high productivity due to high nutrient input and influx of organic matter to the sea floor resulted in low oxygen condition, better preservation of organic matter and favorite environment for deposition of frambooidal pyrite (Fig. 6a). The subsequent intervals are characterized by lower TOC contents, decreased chemical weathering and increased $\delta^{13}C_{\text{org}}$ values, which caused by cooler and drier condition (Fig. 6b). The $\delta^{13}C_{\text{org}}$ values returned to lower values in upper part of the section indicating a normal marine conditions and generally well-oxygenated seafloor after the OAE2. This model refers to the worldwide conditions for the basins during the CTB, which were not influenced by upwelling systems (e.g. Cuba section in the Western Interior Seaway, Elderbak et al., 2014) and can be well defined for the Gharesu and Taherabad sections in the east of Koppeh-Dagh Basin.
In these sections the intervals of warm-humid conditions are coeval with lower $\delta^{18}O$ values and higher TOC. Inversely, the cooler and drier intervals are characterized by higher $\delta^{18}O$ values and lower TOC contents, which can confirm this model (Fig. 4).

**Conclusion**

Global warming, accelerated hydrological cycle and
enhanced marine primary productivity all have been suggested as having contribution to the occurrence of widespread ocean anoxia during the OAE2, but recognizing these factors on a regional scale has remained problematic. In an attempt to separate these forcing factors, we generated geochemical record in a late Cenomanian-early Turonian succession in the east of Koppeh-Dagh Basin laying in the Aitamir-Abderaz formations transition.

However TOC<1% in the study section indicates that the extensive anoxia have not been occurred in the region but higher TOC and TS, increased detrital input and kaolinite/illite ratios (as chemical weathering proxy) in two intervals of the Aitamir Formation indicate that greenhouse condition during OAE2 accelerated chemical weathering and runoff in to the basin. This condition in turn would have led to enhanced nutrient supply and productivity in the basin and either water column stratification.

Increased global burial of organic matter and CO2 consumption due to silicate weathering resulted in falling atmospheric CO2 and provide a relatively cooler and drier conditions, which are characterized by lower TOC values, decreased detrital input and chemical weathering. These cold intervals were also demonstrated by δ13Corg excursion because organisms preferentially take up light carbon12C to produce organic matter. Also, it has been proposed that the rate of isotopic fractionation during carbon fixation by phytoplankton decreases at lower pCO2 values, which led to positive δ13Corg excursion.

The acquiesced data from Hamam-ghaleh section indicate that upper part of the Aitamir Formation was deposited under the OAE2 impressions, but toward the Abderaz Formation the environment returned into the normal marine condition, which provided favorite situation for carbonate production and deposition of marl and limestone of the Abderaz Formation.

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