

## STABLE ISOTOPE ANALYSIS OF THE K/Pg BOUNDARY, SULAIMANIYAH AREA, KURDISTAN, NORTHEAST IRAQ

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Received: 10/ 11/ 2020, Accepted: 01/ 03/ 2021

Keywords: Chemostratigraphy; Stable isotopes; K/Pg boundary; Tanjero Formation;  
Kolosh Formation; Iraq

### ABSTRACT

Stable isotope analysis of oxygen ( $\delta^{18}\text{O}$ ) and carbon ( $\delta^{13}\text{C}$ ) is applied to samples from the Cretaceous – Paleogene boundary (K/Pg boundary) sequence at five localities (Chinarok, Dokan, Dartw, Deramazan, Qalbaza) from Kurdistan Region of NE Iraq. The boundary is located within a thick section of flysch sediments belong to the Tanjero Formation (Maastrichtian) and Kolosh Formation (Paleocene). The purpose of this work is to give an insight into the significance of the isotopes signals in paleoclimatic and paleoenvironmental changes across the boundary zone. Stable isotopes of oxygen ( $\delta^{18}\text{O}$ ) and carbon ( $\delta^{13}\text{C}$ ) were calculated for three samples at each examined locality of a previously well determined K/Pg boundary sequence using nannofossils. The resulted ( $\delta^{13}\text{C}$ ) during Maastrichtian shows negative shift in the samples of the Chinarok, Dartw, and Deramazan localities and the other two localities have positive excursion. This indicate rise of sea water temperature for the first group of samples, i.e. rise of water temperature close to K/Pg boundary. The other two localities have controversial isotopes signals. For the early Danian samples, the Dokan, Deramazan and Qalbaza localities have positive signals with clear increase of the ratio value, the other two localities show positive excursion. This implies a drop of water temperature for the first group which is compatible with global climatic changes, whereas the other two shows contrasted signals. The oxygen isotope ratio ( $\delta^{18}\text{O}$ ) shows similar variable signals among the examined localities. During late Maastrichtian and up to the K/Pg boundary the oxygen isotopes ratio ( $\delta^{18}\text{O}$ ) at four localities (Chinarok, Dokan, Dartw, and Deramazan) show relative drop of value indicating relatively warmer temperature. This drop continue through the early Danian time in two localities (Dokan and Dartw) while the other three have positive excursion suggesting cooler sea water. The heterogeneity of the isotopes signals among the studied localities could be explained in term of environmental effects alterations such as diagenesis or other sedimentation processes. Additional possible explanation is related to the variable sampling spacing at each locality, which could generate mismatching results during correlation.

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## تحليل النظائر المستقرة للحد الفاصل بين الكريتاسي والباليوجيني في منطقة السليمانية، كردستان، شمال شرق العراق

باسم القيم، سوران خرجياني و شيرود وايز

### المستخلص

تم استخدام تحليل النظائر المستقرة للأوكسجين ( $\delta^{18}\text{O}$ ) والكربون ( $\delta^{13}\text{C}$ ) لنماذج صخرية ملتقطة من طبقات الحد الفاصل بين الكريتاسي والباليوجيني من خمس مواقع (جناروك، دوكان، دارتو، ديرمضان وقلبزه) في كردستان، شمال شرق العراق. يفصل هذا الحد بين طبقات الفليش لتكوين التانجيرو (الماسترختي) وطبقات تكوين الكولوش (البالوسيني). يهدف البحث الى التحقق من التغيرات البيئية والمناخية القديمة في طبقات حد التماس المذكور. احتسبت نسبة نظائر الكربون ( $\delta^{13}\text{C}$ ) ونظائر الأوكسجين ( $\delta^{18}\text{O}$ ) في ثلاثة نماذج من كل مقطع من المقاطع المدروسة والتي تم تحديد الحد الفاصل فيها مسبقا بواسطة المتحجرات الدقيقة. تظهر نتائج تحليل نظائر الكربون خلال الماسترختي جنوبا نحو القيم السالبة في مقاطع جناروك ودارتو وديرمضان أما المقطعين الآخرين فكان الجنوب نحو القيم الموجبة مما يشير الى ارتفاع في درجة حرارة المياه للمواقع الثلاثة ونتائج معكوسة للموقعين الآخرين. اما خلال فترة الدانيان المبكر فقد تميزت النتائج في مقاطع دوكان، ديرمضان وقلبزه بانحراف موجب بسبب انخفاض درجات حرارة المياه مقابل انحراف موجب للمقطعين الآخرين بخلاف المقاييس العامة المعروفة. اما نتائج تحليل نظائر الأوكسجين والتي تؤثر بشكل مباشر للتغيرات المناخية، فقد بينت نماذج أربع مقاطع هي جناروك، دوكان، دارتو، وديرمضان لفترة الماسترختي المتأخر الى انخفاض القيم مما يشير الى ارتفاع درجات حرارة المياه مع استمرار هذا الارتفاع الى الجزء الأسفل من الدانيان في مقطعين هما دوكان ودارتو. اما المقاطع الثلاثة الأخرى فقد كانت نتائج التحليل لنسبة النظائر موجبة خلال الدانيان مما يشير الى انخفاض درجة حرارة المياه وهو ما يتوافق مع النتائج الدولية لهذه الفترة. التناقض في بعض النتائج يمكن أن يفسر في ضوء التغيرات البيئية والتحويرية اللاحقة او بسبب اختلاف المسافات الفاصلة بين النماذج مما يظهر تباين في النتائج أثناء المقارنة.

### INTRODUCTION

Stable isotopes of marine sediments provide long continuous records of past climate changes enabling insights into past changes within both oceanic and continental environments. Stable isotopes provide paleoceanographers with the means to reconstruct a range of variables including surface and deep ocean circulation patterns, sea surface and bottom water temperature, sea surface salinity, upwelling intensity and productivity (Maslin and Swann, 2005).

Investigations of stable isotope ratios of carbon and oxygen are a powerful tools for the reconstruction of palaeoenvironmental parameters (Latal *et al.*, 2004), stratigraphic correlation (Korti *et al.*, 2009), and paleoclimates interpretation (Steig *et al.*, 2017). Abrupt changes (excursions) in the stable isotope ratios of minerals and organic matter in ocean sediments and other terrestrial materials are used as stratigraphic markers, indicators of ocean productivity and atmospheric chemistry (Sharp, 1997).

Several studies were reported to use stable isotopes in the evaluation of the K/Pg boundary such as Keller and Lindinger (1989) from El Kef stratotype section in Tunisia; Minoltti *et al.*, 2005 in Bidart section from France; Sepulveda *et al.* (2019), correlation of four sections from Tunisia, Spain, France, and Denmark; Sail *et al.* (2019) for sections from Denmark, Italy, India and Brazil. In addition to Maruoka *et al.*, 2007 and Therrien *et al.*, 2007; for general review of the K/Pg boundary chemostratigraphy.

The K/Pg boundary in the selected localities of this study, was previously defined using calcareous nannofossils biostratigraphy (Kharajiany *et al.*, 2018; Kharajiany *et al.*, 2019; Kharajiany *et al.*, 2020; and Al-Qayim *et al.*, 2020) (Fig.1). The boundary strata are represented by a lower unit of siliciclastic sediments of the Tanjero Formation (Maastrichtian). It is generally characterized by gray to olive gray silty shale, alternate with

medium bedded sandstone, and few limestone horizons. The upper unit is characterized by the siliciclastic sediments of the Kolosh Formation (Paleogene), which is represented by dark gray silty shale alternating with thin sandstone beds and few conglomerate lenses. Both units represent the flysch facies of the Foredeep Zone of the Zagros Foreland Basin (Al-Qayim *et al.*, 2012).

The K-Pg boundary in northern Iraq is long known as a debatable issue with no certain determination of the stratigraphic nature of this important geologic contact. Old studies using planktonic foraminifera biostratigraphy suggest an unconformable nature to this boundary (Bellen *et al.*, 1959; Qassab, 1978; Qassab *et al.*, 1986; Al-Shaibani *et al.*, 1986; Al-Qayim and Al-Shaibani, 1989; Al-Omari, 1995). Recent studies, however, using high resolution biostratigraphic analysis show that this boundary is conformable with no definite gap (Sharbazheri, 2007; Mousa *et al.*, 2020; and Bamerni *et al.*, 2020). A detailed biostratigraphic study using calcareous nannofossils from seven localities of Sulaimaniyah area of northeast Iraq shows that the K/Pg boundary at these localities is conformable (Kharajani, 2019). The boundary at these localities is located between the Tanjero Formation (Maastrichtian) with recognition of a complete CC26 biozone, and the Kolosh Formation (Paleogene) with the occurrence of NP1, NP2 biozones of the Danian (Fig.2). Five of these localities were selected for the stable isotope analysis of this study.

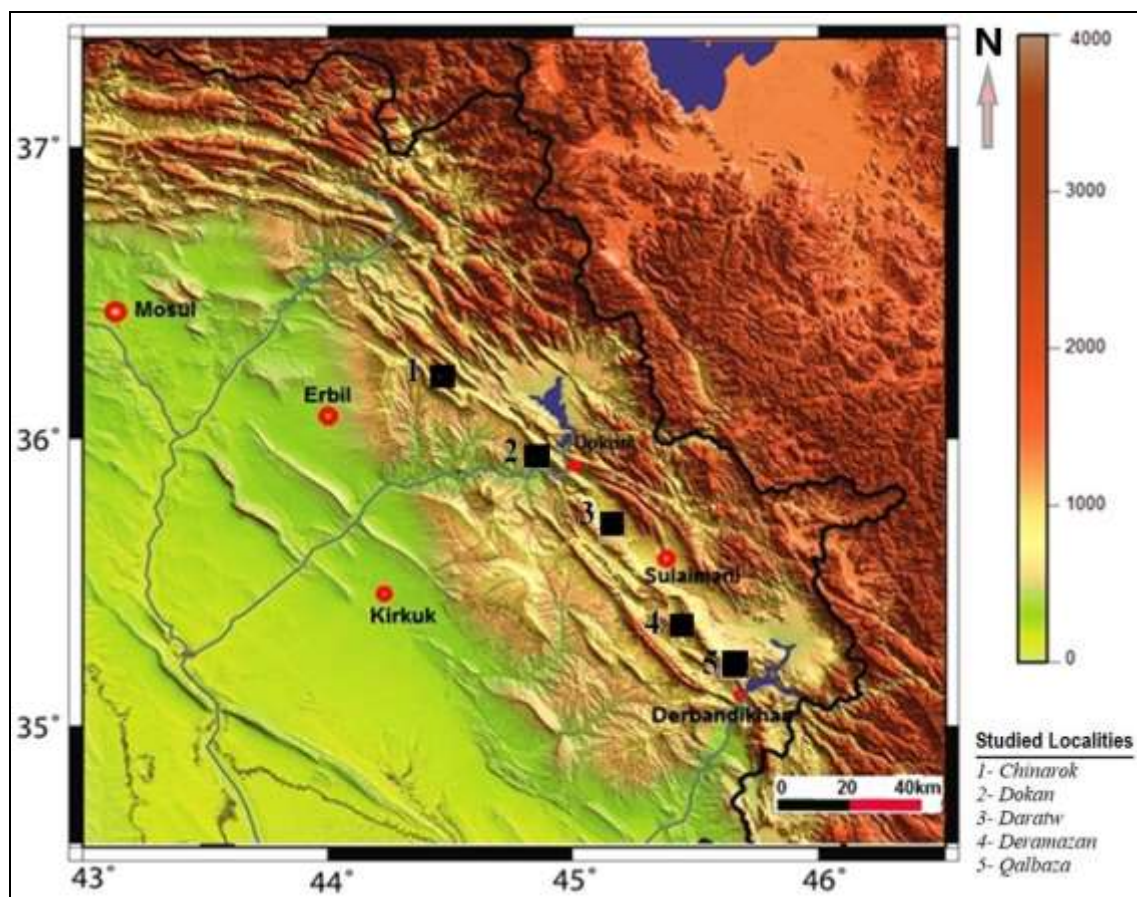


Fig.1: Shaded relief map of northeastern Iraq showing location of the studied sections

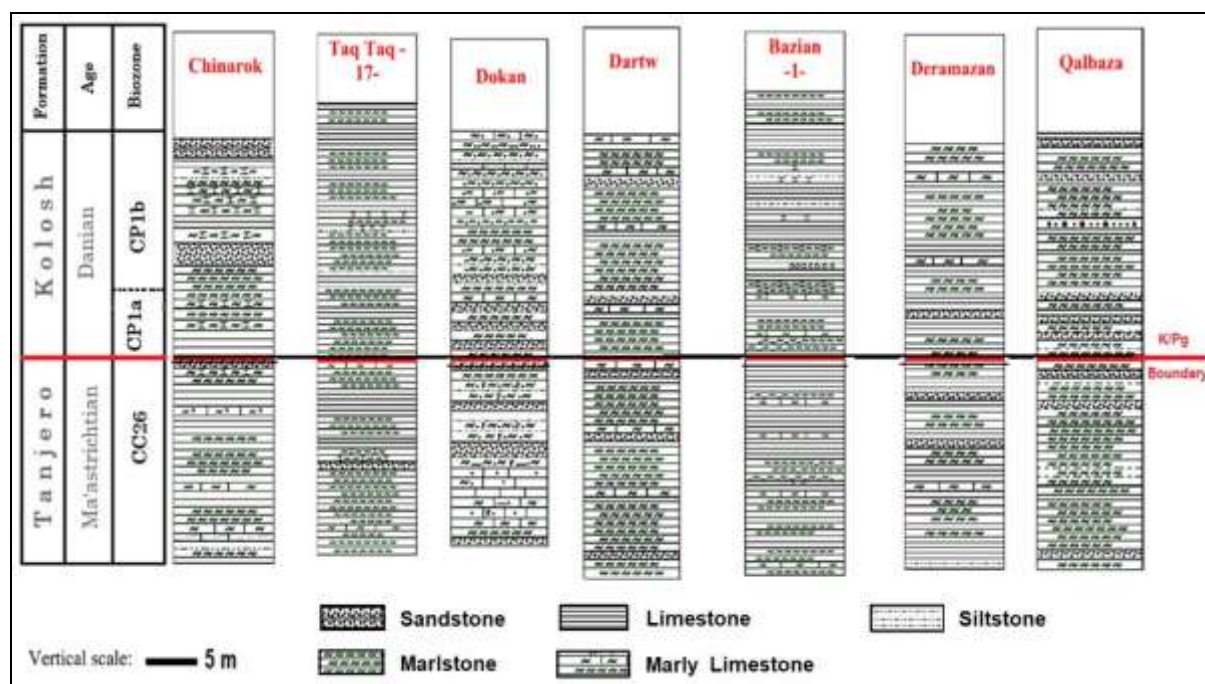


Fig.2: Biostratigraphic correlation of the K/Pg boundary at seven localities in Sulaimaniyah area NE Iraq (from Kharajany, 2019)

## MATERIALS AND METHODS

Five localities are selected from the surroundings of Sulaimaniyah City in the Kurdistan Region of northeastern part of Iraq. All these sections are located at the southwestern margin of the High Folded Zone. The localities are: Chinarak, Dokan, Dartw, Deramazan, and Qalbaza (Fig.1). Three samples were collected from each locality (except Dokan locality has four samples). One sample is taken from the Maastrichtian sediments of the Tanjero Formation. The second sample is collected from the exact position of the K/Pg boundary zone. The third sample is collected from the Danian sediments of the Kolosh Formation.

The selected rock samples were taken from marlstone horizons across the boundary strata of each section. Spacing between samples are not exactly the same for each section, because marly limestone and marlstone rock type are the chosen lithology for whole-rock isotope analysis. The vertical distribution of these lithologies in clastic-dominated sequences is not the same. Each sample is dried, crushed, and grinded to powder size. About 4 – 5 milligrams were taken from each grinded sample and poured into the plastic tube and run in the mass spectrometer. The samples were run on a thermo-isotope ratio mass spectrometer interfaced with a gas bench two-heads pace sampler. The samples were weighed into exetainers flushed with helium, and doped with 105%  $H_3PO_4$  for at least 18 hours. The resulting  $CO_2$  was analyzed. Compounds of pure chemical substances are used to test the ratio values linearity and define instrument response for the determination of elemental composition during sample running. Standard deviation is calculated for the samples identifiers to ensure analysis precision and measurements calibration (Table 2).

Variations in isotope ratios from isotopic fractionation process are measured using mass spectrometry, which separates the different isotopes of an element based on their mass-to-charge ratio (Table 1). To correlate the chemostratigraphic data among studied localities and

to display variation of isotopic ratios of both carbon and oxygen in each studied section, the resulted ratios were plotted against lithology of the K/Pg strata at each locality (Figs.3 and 4).

Table 1: Stable isotope analysis results of the studied samples

Location	Identifier 1 (Sample No.)	Carbon $\delta^{13}\text{C}$	Oxygen $\delta^{18}\text{O}$
Chinarok	CHO51	-6.63	26.64
	CHO43	-1.20	26.32
	CHO29	-0.92	26.92
Dokan	DKN83	-3.34	26.03
	DKN77	-3.71	24.65
	DKN71	+11.23	26.43
	DKN65	+6.39	29.42
Dartw	RTW16	-2.00	25.05
	RTW7	-0.51	25.66
	RTW1	-0.07	25.70
Deramazan	RMZ(-12)	-0.04	26.84
	RMZ(-15B)	-3.18	24.33
	RMZ(-17)	-0.82	26.01
Qlbaza	QLB25	+0.04	28.01
	QLB17	-1.41	27.18
	QLB14	-2.27	26.73

Table 2: Standard samples (Identifiers) used to ensure precision of the isotopes analysis

Identifier	d $\delta^{13}\text{C}$	d $\delta^{18}\text{O}$
23	-0.61	15.59
23	-0.58	15.72
23	-0.54	15.79
St. Dev	0.033307	0.103548
average	-0.58	15.70
actual	-0.6	15.75
Identifier 1	d $\delta^{13}\text{C}$	d $\delta^{18}\text{O}$
NBS-19	1.89	28.62
NBS-19	1.90	28.77
NBS-19	2.00	28.79
St. Dev	0.058979	0.090933
average	1.93	28.73
actual	1.95	28.72
Identifier 1	d $\delta^{13}\text{C}$	d $\delta^{18}\text{O}$
22	-35.65	13.32
22	-35.58	13.35
22	-35.57	13.38
St. Dev	0.041053	0.027729
average	-35.60	13.35
actual	-35.6	13.31

## RESULTS

The ratios of the carbon stable isotope ( $\delta^{13}\text{C}$ ) and oxygen stable isotopes ( $\delta^{18}\text{O}$ ) were calculated according to the standard procedures and the values of these ratios in each sample were tabulated in Table (1). Various identifiers or reference standard ratios for the resulted data of both carbon and oxygen isotopes were used to calibrate measurements, and are shown in Table (2).

These ratios were plotted against the lithology of each section to reveal relations to stratigraphy (Figs.3 and 4). Most of the readings for the carbon isotopes are negatives except two readings from Dokan section and one reading from Qalbaza section are positive. For the oxygen isotopes all the measured values are positive.



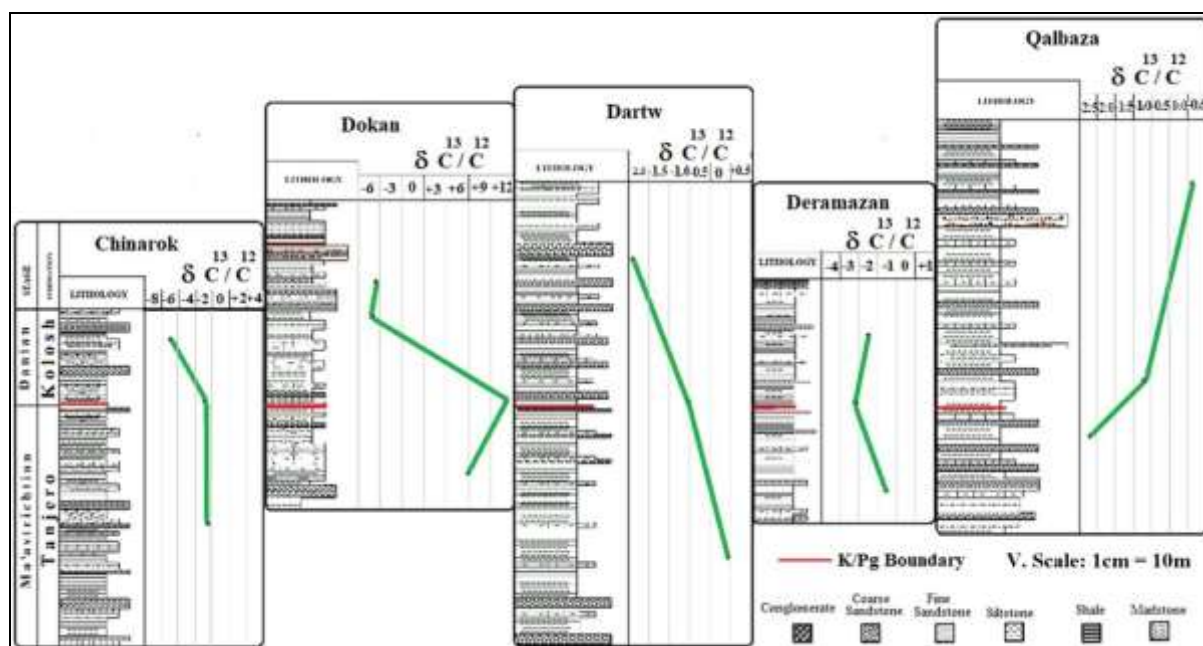


Fig.3: Stable carbon isotopes ratios ( $\delta^{13}\text{C}/^{12}\text{C}$ ) showing vertical variation in the studied samples of the K/Pg strata

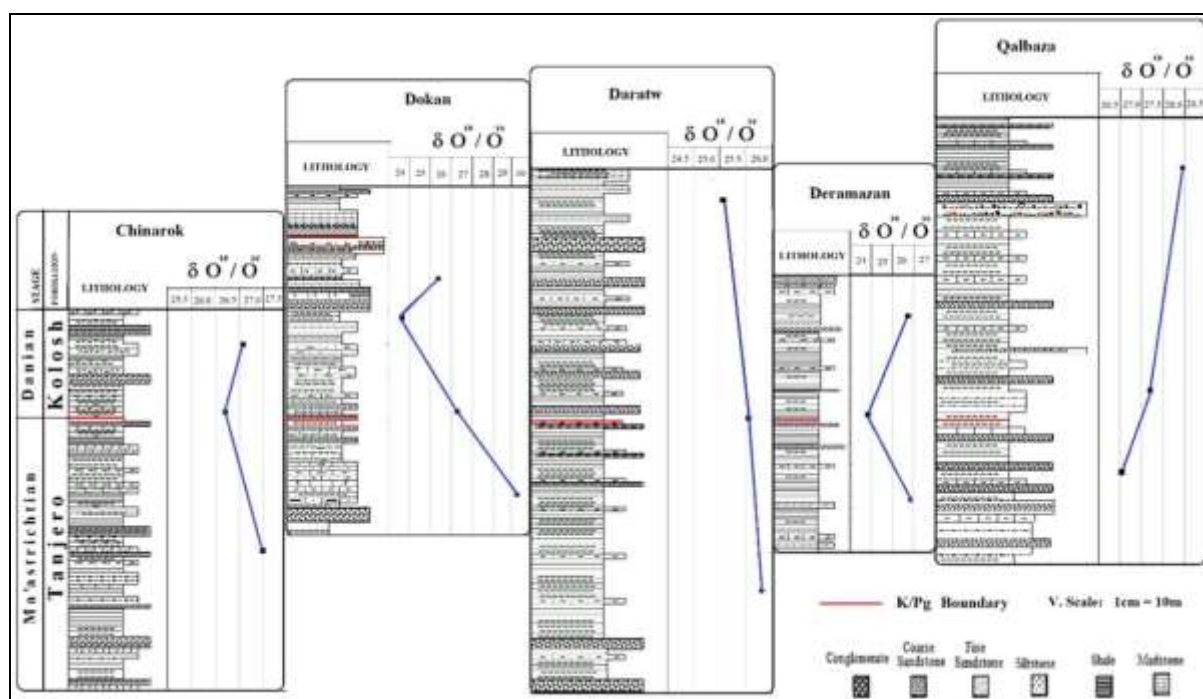


Fig.4: Stable oxygen isotopes ratio ( $\delta^{18}\text{O}/^{16}\text{O}$ ) showing vertical variations in the studied samples of the K/Pg strata

## DISCUSSION

The plotting of the resulted ratios against the lithology shows inconsistent trend of changes among the examined samples and across the K/Pg boundary strata in the studied sections (Figs.3 and 4). This heterogeneity of the isotopes data are probably due the complex

processes that control isotopes signatures such as; the actual value of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  in sea water (Maslin and Swann, 2005); Ocean water temperature (Maslin and Swann, 2005; Keller, 1989; Sharp, 1997; Korti *et al.*, 2009; Sepulveda *et al.*, 2019); diagenesis (Sharp *et al.*, 2000; Minoletti *et al.*, 2005, Schubben and Schootbrugge, 2019), and isotopes fractionation processes (Maslin and Swann, 2005; and Adatte *et al.*, 2002). However, ancient carbonates commonly retain their primary carbon isotopic composition (Marshall, 1992; Kaufman and Knoll, 1995; Knoll *et al.*, 1995 in Nagarajan *et al.*, 2008). Therefore interpretation of the isotopes data for the examined sections is conceivably over generalized and would be of limited applications. Additionally, the limited number of the examined samples at each section overshadows the reliability of the observed signals. However, the low number of the studied samples is justified by the purpose of the study which is to characterize the specific strata of the K/Pg boundary rather than characterizing the whole formation isotopic composition.

▪ **Carbon isotope analysis ( $^{13}\text{C}/^{12}\text{C}$  ratio)**

The noticeably clear variation for the ( $^{13}\text{C}/^{12}\text{C}$ ) isotopes ratio or ( $\delta^{13}\text{C}$ ) in samples from all studied sections (Fig.3) is quite normal due to the joint effect of many geochemical and physical parameters as mentioned above. Comparison of the ( $\delta^{13}\text{C}$ ) readings during the Late Maastrichtian towards K/Pg boundary show shifting between negative and positive values. The analyzed samples of Chinarok, Dartw and Deramazan localities show shifting of ratio value towards negative side, whereas values in Dokan and Qalbaza localities move towards the positive excursion. During the early Danian time the analyzed samples show relatively a clear increase of the ratio value or positive shift of ( $\delta^{13}\text{C}$ ) in samples from the at Dokan, Deramazan, and Qalbaza localities (Fig.3). The other two localities however have slight negative shift.

The drop of the carbon isotope ratio ( $\delta^{13}\text{C}$ ) towards the K/Pg boundary zone can be taken as an indication of possible rise in sea water temperature during the transition from upper most Maastrichtian to the K/Pg boundary Zone (Maruok *et al.*, 2007; Keller, 1989; Sharp, 1997; Korti *et al.*, 2009; Sepulveda *et al.*, 2019). Therrien *et al.* (2007), noticed that the values of ( $\delta^{13}\text{C}$ ) are the lowest within 6 cm. above the K/Pg, then it return back to its pre-boundary level. Similarly Sepulveda *et al.* (2019) noticed that the ( $\delta^{13}\text{C}$ ) drops down during transition across the very boundary strata of four selected localities including the K/Pg Global Stratotype locality of El Kef at Tunisia (Fig.5)

During the Early Danian time the values of the ( $\delta^{13}\text{C}$ ) ratio is increased and show shifting towards positive readings at Dokan, Deramazan, and Qalbaza localities (Table 1 and Fig.3). This is interpreted as cooling back of the sea water after crossing the boundary (Keller, 1989; Therrien *et al.*, 2007; and Sepulveda *et al.*, 2019). The other two localities (Chinarok and Dartw) show opposite excursion (Fig.3). These anomalous readings are possibly related to local diagenetic effects or other possible environmental effect as mentioned above. However, the diagenetic effect is considered limited here because the samples are mainly of marlstone rock type which shows ineffective diagenetic alterations (Kharajiani *et al.*, 2019). The other explanation of these confusing results at these two sections may be related to the sampling spacing differences at each of the studied sections, which are not uniform. Such irregular spacing of the collected samples could lead to the missing of the proper values at correlatable strata due to the rapid fluctuation of the ( $\delta^{13}\text{C}$ ) values around the K/Pg boundary zone.

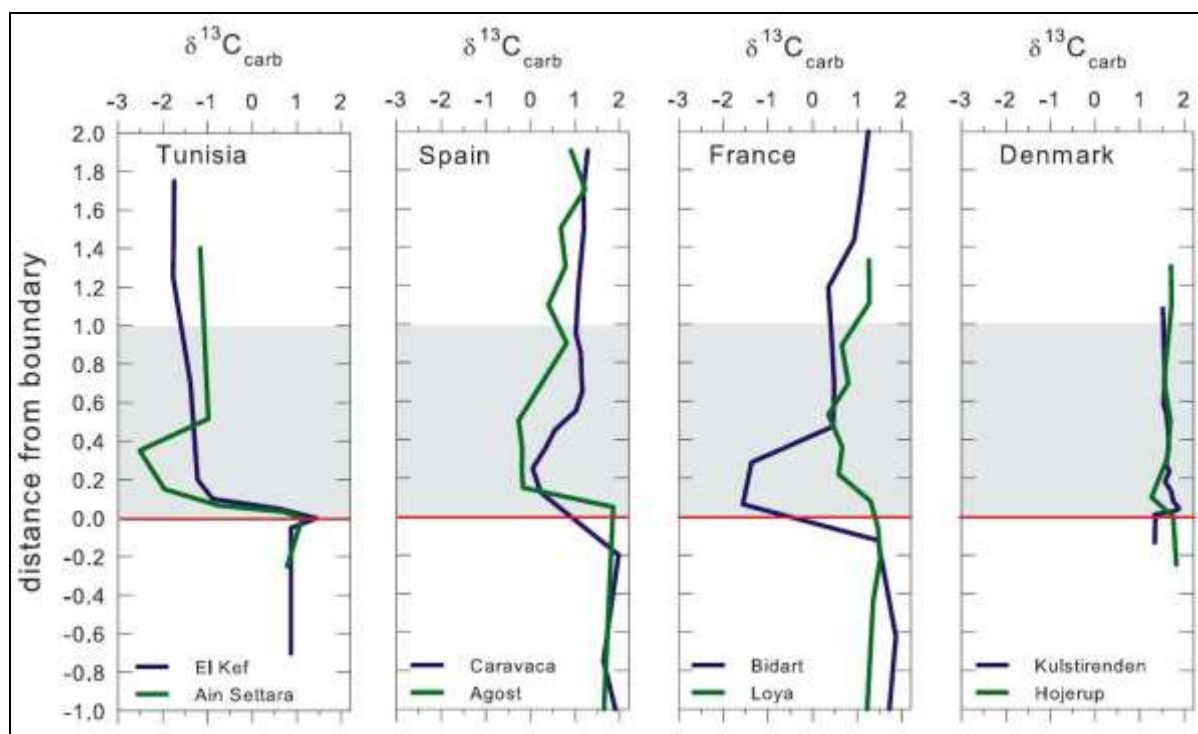


Fig.5. Comparison of carbon stable isotope signature ( $\delta^{13}\text{C}$ ) among different sections of the world. Gray area represents the position of the K/Pg boundary clay layer at each location. (From Sepulveda *et al.*, 2019)

#### ▪ **Oxygen isotope analysis** ( $^{18}\text{O}/^{16}\text{O}$ ratio)

The  $^{18}\text{O}/^{16}\text{O}$  ratio or ( $\delta^{18}\text{O}$ ) isotopes changes over the ages and these changes are a function of changes in climate condition namely temperature (Jouzel *et al.*, 1994). Three major factors affect the marine oxygen isotope record. The first is the water temperature at which the sediments were precipitated. As ocean water warms,  $\delta^{18}\text{O}$  ratio in the inorganic precipitates is lowered. Second is the actual  $\delta^{18}\text{O}$  of the water ( $\delta^{18}\text{O}_w$ ) in which the inorganic precipitate are produced. The third factor which controls the  $\delta^{18}\text{O}$  is the amount of isotopically light freshwater that is stored in ice sheets (Maslin and Swann, 2005). Additional factors that affect  $\delta^{18}\text{O}$  ratio of sea water is the local evaporation, which causes  $^{18}\text{O}$  enrichment, and freshwater input from precipitation or coastal runoff, which causes  $^{16}\text{O}$  depletion (Sharp *et al.*, 2000).

The calculated oxygen isotope data were correlated among the studied sections to show variable changes across the boundary zone (Fig.4). It is obvious that the Chinarok, Dokan, Dartw, and Deramazan sections show a general trend of decrease in values of oxygen isotopes ratio ( $\delta^{18}\text{O}$ ) from latest Maastrichtian to the K/Pg boundary zone (Fig.4). This decrease is interpreted to be related to rise in temperature near K/Pg transition (Keller, 1989; Maslin and Swann, 2005, Table 3). Similar inference is drawn from the ( $\delta^{13}\text{C}$ ) data of the same localities except Dokan locality.

During The Early Danian time, and after crossing the K/Pg boundary zone, the Chinarok, Deramazan, Qalbaza and (to certain extent) Dokan localities, show that the oxygen isotopes ratio ( $\delta^{18}\text{O}$ ) is shifted towards positive values (Fig.4). This might be explained as a drop of sea water temperature and a beginning of a cooling episode (Keller, 1989; Maslin and Swann, 2005, Table 3). Epstein *et al.* (1953) explained that an increase of the oxygen concentration is



directly related to a decrease of the temperature, by means of a complex mechanism of evaporation/ condensation of the ocean-atmosphere system.

Table 3: Environmental influence on marine oxygen isotopes  
(From Maslin and Swann, 2005)

Environmental Factor	Increase	Decrease
Temperature	$\delta^{18}\text{O}$ decrease	$\delta^{18}\text{O}$ increase
Global Ice Volume	$\delta^{18}\text{O}$ increase	$\delta^{18}\text{O}$ decrease
Salinity	$\delta^{18}\text{O}$ increase	$\delta^{18}\text{O}$ decrease
Density	$\delta^{18}\text{O}$ increase	$\delta^{18}\text{O}$ decrease

The Dartw locality shows continuation of decreasing trend of ( $\delta^{18}\text{C}$ ) values which imply a warming episode. This result contradicts the trend in the other four localities. This is more likely to be related to either diagenetic effect which is less possible for marlstone rock type, or due to mismatching of correlative readings by irregular sampling intervals of highly fluctuated isotope ratio zone among the studied sections. In fact even in some international standard measurements of representative localities, these dissimilarities can be also noticed (Fig.5). This variability might be explained in term of various environmental effects including diagenesis or sedimentation processes. The other explanation is related to sampling spacing differences at each of the examined section, which could lead to mismatching of the correlative stratigraphic horizons of the similar isotopic signals. This makes these results to have indirect application in locating the K/Pg boundary. However it is safe to say that the vertical changes across the K/Pg transition of both isotopes (oxygen and carbon) in samples from Dokan, Deramazan, and Qalbaza shows similar vertical variability (Figs.3 and 4) and are compatible with global trends.

## CONCLUSIONS

Stable isotopes analysis of carbon  $\delta^{13}\text{C}$  and oxygen  $\delta^{18}\text{O}$  ratios for samples selected across the K/Pg boundary at five localities in northern Iraq reveal heterogeneous signals at the boundary sequence. However, an overgeneralized conclusion drawn from three localities (Dokan, Deramazan, and Qalbaza) out of five localities shows compatibility with global variations of an increase of temperature towards the K/Pg boundary strata. The samples of the Lower Danian at these three localities shows drop of temperature and returning back to the cooling stage. The anomalous values of the other two localities are believed to be related either to the effect of other environmental factors, such as diagenesis or sedimentary processes, or due to the collected sample spacing which is not uniform. The heterogeneity among the resulted data makes the use of the analyzed isotopes in locating the K/Pg boundary less applicable in the investigated sections.

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