

Clumped isotope record of individual limestone fabrics: A potential method to constrain the timing of oil migration

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ABSTRACT

This study applied clumped isotope analyses to investigate how different limestone components (larger skeletal grains and enclosing matrix) and cements may have varying degrees of susceptibility to recrystallization during progressive burial. The results also provide new constraints on the temperatures of recrystallization and cementation, the nature of the waters involved, the timing of the diagenetic events, and the effect of oil emplacement on inhibiting diagenetic processes. We used clumped isotope measurements from core samples in two giant oilfields, together with petrography and well-constrained thermal histories, to study the reactivity of different limestone fabrics and whether the displacement of pore water by oil affected the recrystallization processes. We recognize seafloor micritization, cementation, and recrystallization as distinct diagenetic processes. The results indicate that skeletal grains record burial temperatures of 60 °C to 72 °C and enclosing coarse-blocky calcite cements record temperatures of 85 °C to 86 °C. Oxygen isotopes suggest that both processes involve high water/rock ratios. Burial histories together with carbonate Δ_{47} values are used to estimate that the skeletal grains recrystallized from 99 to 70 Ma and the cements formed at around 54 Ma. By contrast, the matrix shows temperatures of 68 °C to 90 °C, consistent with continuous recrystallization at low water/rock ratios from 90 to 50 Ma. The large skeletal grains thus tend to recrystallize early, but are less reactive during subsequent burial because of larger crystal size, whereas the more finely crystalline and reactive matrix more faithfully records maximum burial conditions. The lower temperature recorded in the matrix relative to the present reservoir temperatures is interpreted as reflecting stoppage or inhibition of recrystallization by oil emplacement.

1. Introduction

Carbonate rocks provide a large proportion of the world's reservoirs for hydrocarbons and water, and are thus important for energy resources and access to clean water. However, to contain fluids, rocks need porosity, and carbonate porosity evolution is complex because of the high chemical reactivity of the carbonate minerals (Choquette and Pray, 1970; Moore, 1989a). The carbonate reservoir quality depends on the diagenetic process within the porosity, especially during burial. Essentially, all of the diagenetic reactions during burial diagenesis are aqueous dissolution-(re)precipitation reactions, and it has long been suggested that displacement of water by petroleum could inhibit or stop these reactions (Feazel and Schatzinger, 1985). The theory is that the petroleum coats on the mineral inhibits the surface reactions required for the dissolution and precipitation process as most minerals are insoluble in hydrocarbon fluids (Feazel and Schatzinger, 1985; Neilson et al., 1998). This has been conclusively demonstrated to occur only in

microporous limestone reservoirs, based on trends of increasing porosity towards the crests of domal structures within strata having little lateral variation in depositional texture (D'Heur, 1984; Oswald et al., 1995; Neilson et al., 1996; Brasher and Vagle, 1996; Melville et al., 2004; Ehrenberg et al., 2016; Van Simaey et al., 2017; Ehrenberg et al., 2020). Reports of inferred porosity preservation by petroleum in other types of carbonate hosts are unconvincing because variations in depositional texture are not considered, and interpretations of paleo-oil/water contact position are open to doubt. Sandstone reservoirs also show no evidence of any petroleum emplacement effect (i.e. quartz cement retardation) (Giles et al., 1992; Bjorkum and Nadeau, 1998; Molenaar et al., 2008), despite clear evidence that porosity in the oil zone of certain Upper Jurassic sandstones has been preserved by grain-coating microquartz (Aase et al., 1996). The cement retardation process on both sandstone and carbonate reservoir is similar in theory: the displacement of pore water by oil prevents or significantly retards the precipitation of cement as most minerals are insoluble in hydrocarbon

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fluids (Feazel and Schatzinger, 1985; Neilson et al., 1998). However, there has to date been little examination of the detailed timing and geochemistry of processes by which petroleum inhibits diagenesis in microporous limestone reservoirs, and this is the main focus of the present study.

Carbonate burial diagenesis is controlled by the mineral-surface area available for reaction, the saturation state of diagenetic fluids, and the thermodynamic stability of the carbonate minerals being dissolved or precipitated (Flügel, 2010). The fluids are commonly chemically complex brines resulting from long-term water-rock interaction under elevated temperatures and pressures (Stoessell and Moore, 1983; Moore, 1989b, 2001). At the same time, elevated burial temperatures increase the rates of chemical reactions and ionic diffusion (Moore, 2001). Therefore, mass transfer reactions expressed by dissolution and precipitation (recrystallization) (Stolper and Eiler, 2015) may occur during burial. In the subsurface conditions, the recrystallization process of the rocks records the ambient burial temperature and, depending on the abundance of diagenetic fluid in the pore system, the bulk isotopic values ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) may be altered (Dale et al., 2014; Beckert et al., 2016; Lawson et al., 2018; Bergmann et al., 2018; Ryb and Eiler, 2018; Stolper et al., 2018; John, 2018; Ingalls, 2019; Veillard et al., 2019; Lukoczi et al., 2020; Ryb et al., 2021). Comparison between the temperatures recorded in recrystallized carbonates and established burial histories and the timing of petroleum accumulation provides a way of testing the hypothesis that water displacement inhibits or stops diagenesis.

The issue to date is that established proxies to estimate recrystallization temperatures (notably fluid inclusions) are difficult or impossible to apply in carbonates with very small crystal sizes (i.e., micrite) (Mathieu et al., 2013; Mraz et al., 2019). Here, we used carbonate-clumped isotope paleo-thermometry to circumvent this problem. This is a recently developed technique for estimating the temperature of the formation of carbonate minerals based on the proportion of ^{13}C and ^{18}O atoms bonded together in carbonate ions within the mineral lattice (Ghosh et al., 2006). This technique can be applied without knowing the isotopic composition of the diagenetic water (Ghosh et al., 2006) and thus provides both paleo-temperature and fluid composition independently of each other. Clumped isotopes have been applied to thermal and burial history reconstruction (Dennis and Schrag, 2010; Passey and Henkes, 2012; Henkes et al., 2014; Huntington and Lechler, 2015; Shenton et al., 2015; Gallagher et al., 2017; Ryb et al., 2017; John, 2018; Manganot et al., 2018), diagenesis (Huntington et al., 2011; Loyd et al., 2012; Dale et al., 2014; Millan et al., 2016) and subsurface fluid reconstruction (Budd et al., 2013; Lu et al., 2017; Lu et al., 2018).

From previous studies (Shenton et al., 2015; John, 2018; Veillard et al., 2019) we know that the clumped isotope technique is sensitive to complete and partial recrystallization and thus yields information on diagenetic processes. The previous body of work also reported that each carbonate fabric has a different susceptibility to dissolution-reprecipitation (Shenton et al., 2015). Here, we use the clumped isotope technique on the fine-grained matrix, skeletal grains, and cements of subsurface carbonates from an oil-bearing reservoir in the Arabian Plate. This location is an ideal study location because of previous work on the diagenetic and thermal history using fluid inclusions and petrography (Burruss et al., 1985; Alsharhan, 1989; Oswald et al., 1995). The same studies also suggested that this interval experienced multiple recrystallization processes, implying the possibility of over-printed fluid inclusions in the carbonate minerals. Our objectives are a) to further understand the response of different limestone fabrics to thermal resetting with respect to clumped isotopes, and b) to investigate whether carbonate diagenesis (cementation and recrystallization) continued after hydrocarbon emplacement, as has been debated (Neilson et al., 1996; Neilson et al., 1998; Barclay et al., 1999; Cox et al., 2010; Kolchugin et al., 2016).

2. Geological setting of the carbonate interval

The sedimentary system in the study area was initially filled by the prograding of the Lekhwair/Habshan/Salil (LHS) system during the Barremian age (Landmesser and Saydam, 1996; van Buchem et al., 2002) followed by aggradation of the Kharaiib, Shuaiba, Nahr Umr and Natih formations (van Buchem et al., 2002). The Barremian-Aptian interval comprises the Shuaiba, Kharaiib, and Lekhwair formations, which developed on a broad carbonate ramp (van Buchem et al., 2002; van Buchem et al., 2010; Vahrenkamp et al., 2014). The Barremian strata in our study (Fig. 1) comprise sequences I and II (van Buchem et al., 2002), which include three early facies associations: 1) orbitoloid/miliolid wackestone to packstone, 2) mixed-fauna mudstone with wackestone, and 3) rudist/miliolid wackestone, packstone, grainstone, and frame-stone (van Buchem et al., 2002).

3. Methods

3.1. Sampling

Samples for this study come from cores collected in seven wells from an onshore giant oilfield and one well from an offshore giant oilfield. The samples are limestones from the uppermost of two major Barremian-age reservoirs. Small amounts of different textural origins, including skeletal grains, matrix, and cement, were extracted from cut surfaces using a dental drill at the lowest possible speed (1000–2000 rpm). To obtain a homogeneous powder size, the samples were subsequently gently crushed with an agate mortar and pestle and then sieved through a 125 μm sieve. Because of the presence of oil residue, the offshore field core samples were treated with low-temperature plasma ashing (Henniker Plasma HPT-100) for 2×10 min duration (Adlan et al., 2020) under oxygen at a flow rate of 46 mL/min and a power of 100 W, with a vacuum condition of 0.2 mbar. This plasma treatment does not affect the original isotopic composition of the sample (Adlan et al., 2020).

Age		Lithostratigraphy Units		3 rd order Sequence	Lithology
Lower Cretaceous	Aptian	Shuaiba Fm.		Aptian IVa	Basinal Limestones
				Aptian III	
				Aptian II	
				Aptian I	
	Barremian	Kharaiib Fm.		Hiatus	Shallow-water Limestone
				Barremian II	
				Barremian I	
		Lekhwair			Argillaceous Limestones

Fig. 1. Carbonate formations used for this study with stratigraphic units modified from Van Buchem et al., (2010). Samples were obtained from carbonate reservoir rock of the lower Cretaceous Upper Thamama Group, Aptian-Barremian (Barremian II sequence) of two giant oilfields on the Arabian Plate located onshore and offshore each.

3.2. Petrography and mineralogy

Petrographic observations were made using a transmitted light Zeiss Axioskop 40, followed by cathodoluminescence (CL) microscopy on a Nikon Eclipse 50i microscope equipped with a CITL cathodoluminescence MK5–2 stage. The operating conditions for the CL stage were a current of 315 μ A, an accelerating voltage of 15 kV, and a vacuum operating at 0.003 mbar. Fourier Transform Infrared Spectroscopy (FT-IR-Spectrometer Nicolet 5700) was used to ensure the samples were homogenous and to qualitatively estimate mineralogy (calcite) following Henry et al. (2017).

3.3. Stable isotope measurement

Clumped isotopes, $\delta^{18}\text{O}$, and $\delta^{13}\text{C}$ measurements were carried out in the Qatar Stable Isotope Laboratory at Imperial College London using our fully automated IBEX (Imperial Batch EXtraction) system. A total of 4 mg of calcite powder per replicate analysis was preloaded on a 40-positions carousel, and each replicate analysis was individually dropped in a vacuum-sealed common acid bath for reaction with 105% orthophosphoric acid at 90 °C for 10 minutes (McCrea, 1950; Swart et al., 1991). This acid digestion generates CO_2 which flows through a series of water traps, silver wool and Porapak-Q trap (Huntington et al., 2009; Davies and John, 2019) to remove water, sulphur, and hydrocarbons. The resulting purified CO_2 was then automatically loaded and analysed on a dual inlet Thermo MAT 253 mass spectrometer (Thermo Instrument Bremen, Germany) modified with Farraday collectors registered for m/z 44–49. The measurements were conducted using a reference High Purity (HP) CO_2 gas with isotopic values of $\delta^{18}\text{O}$ VSMOW 8.6‰ and $\delta^{13}\text{C}$ VPDB -36.8‰. Each replicate measurement comprises eight acquisitions with seven cycles each of 26 s integration time (John and Bowen, 2016). A minimum of three replicate analyses were acquired per sample, with a total analysis time of 2 h per sample. The uncertainty of the isotopic measurements in this study is reported by 1 standard error.

3.4. Data analysis and reduction

Calculations and corrections of raw Δ_{47} , $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ were performed using the free software Easotope (John and Bowen, 2016). Non-linearity in the mass spectrometer was corrected with the pressure baseline correction methods developed at ETH Zurich (Bernasconi et al., 2013), and the Δ_{47} results were projected in the inter-laboratory absolute reference frame or Intercarb Carbon Dioxide Equilibrated Scale (I-CDES) (Bernasconi et al., 2021) based on our routinely measured ETH1, ETH2, ETH3, ETH4 and our internal Carrara marble (ICM) carbonate standards (Muller et al., 2017; Davies and John, 2019; Bernasconi et al., 2021). This Δ_{47} (I-CDES) was then used to calculate a the temperature following a recent multi-lab calibration (Anderson et al., 2021). The standards-to-samples ratio used in our study is one standard per three sample measurements. The bulk isotopic value of $\delta^{18}\text{O}$ was corrected for acid digestion at 90 °C by multiplying the value by 1.0081 using the published fractionation factor (Kim et al., 2007). Mass 48 and mass 49 signal spikes were monitored for each measurement to detect hydrocarbons, chlorocarbons (Eiler and Schauble, 2004; Huntington et al., 2009) and sulphur-bearing contaminants (Guo and Eiler, 2007; Huntington et al., 2009). To be deemed clean, a replicate analysis needed a Δ_{48} offset value <1.5‰ and/or a 49 parameter value <0.3 (Davies and John, 2017).

4. Results

4.1. Petrography

The textures of the analysed samples are described in Table 1. The major allochems found in the samples were peloids, miliolids, and mollusc fragments. The rudists found in the samples have been

Table 1

Sample descriptions.

Well Identifier	Depth (m)	Petrographic Description	Component Analysed	Sample ID
<i>Onshore Giant Field</i>				
Ibrahim-911	2719	Packstone to wackestone, rudist, orbitolinid and foraminifers chamber filled with cement	Matrix (Mic _x)	K89220_m
			Skeletal (Rud)	K89220_sk
Ibrahim-9139	2743	Packstone to wackestone, blocky calcite cement	Matrix (Mic _x)	L90004_m
	2745	Rudist (Glossomyophorus costatus) skeletal floatstones with micritized packstone matrix, blocky calcite cement with micrite inclusion	Matrix (Mic _x)	L90088_m
			Skeletal (Rud)	L90088_sk
			Cement (Cm)	L90088_cem
Ibrahim-919	2823	Packstone to wackestone, blocky calcite cement	Matrix (Mic _x)	M92641_m
	2829	Floatstones with packstone to wackestone matrix	Skeletal (Rud)	M92836_sk
Ibrahim-922 ^a	2963	Packstone to wackestone with blocky calcite cement, peloids and miliolids fragments	Matrix (Mic _x)	N97205_m
			Skeletal (Rud)	N97205_sk
Ibrahim-926 ^b	2918	Grainstone to packstone with blocky calcite cement, miliolids and molluscs fragments	Matrix (Mic _x)	W957510_m
			Skeletal (Rud)	W957510_sk
Ibrahim-973	2760	Packstone, forams chamber filled bwith cement	Matrix (Mic _x)	X90571_m
	2764	Rudist skeletal floatstones with packstone to wackestone matrix, equant/blocky calcite cement along with minor dolomite crystal	Matrix (Mic _x)	X90704_m2
			Matrix (Mic _x)	X90704_m1
			Cement (Cm)	X90704_cem
Ibrahim-980	2746	Rudist skeletal floatstones with packstone to wackestone matrix, micritized rudist, blocky calcite cement	Matrix (Mic _x)	Y90101_m2
			Matrix (Mic _x)	Y90101_m1
			Mic. Skeletal (Rud _m)	Y90101_sk (mic)
<i>Offshore Giant Field</i>				
Rainy-9316	2886	Floatstones with micritized packstone matrix, equant/blocky calcite and minor dolomite cement	Matrix (Mic _x)	Z94708_m
			Skeletal (Rud)	Z94708_sk

^a Sample obtained 63 m below the free-water level (100% water saturated).

^b Sample obtained 18 m below the free-water level (100% water saturated).

previously reported (Ehrenberg et al., 2018) as *Glossomyophorus costatus* (Masse et al., 2015) and *Agriopleura* cf. *libanica* (Astre) (Masse and Fenerci-Masse, 2015).

The term ‘skeletal grain’ refers to >2 mm skeletal fragments that could be sampled using a dental drill. The term ‘matrix’ refers to the grain-supported packstone matrix, including both allochems and recrystallized mud. (Figs. 2–4). The term ‘micritization’ is used to refer to both the breakdown of the original skeletal to form micrite on the seafloor (Reid et al., 1992; Reid and Macintyre, 1998) and the microborings infilled by micrite (Bathurst, 1966). The matrix of our predominantly wackestone and packstone samples consists of micritized skeletal fragments/foraminifers (“Mic_x” and “F”, Fig. 2.C). This micritization is typically associated with seafloor processes (Reid et al., 1992; Reid and Macintyre, 1998). Other micritization signatures identified in our thin sections are boring infilling (“Bor”) within rudist shell fragments (“Rud”, Fig. 3.E₁ and Fig. 4.H₁). The precipitation of micrite in open borings is classically interpreted as generated by organisms

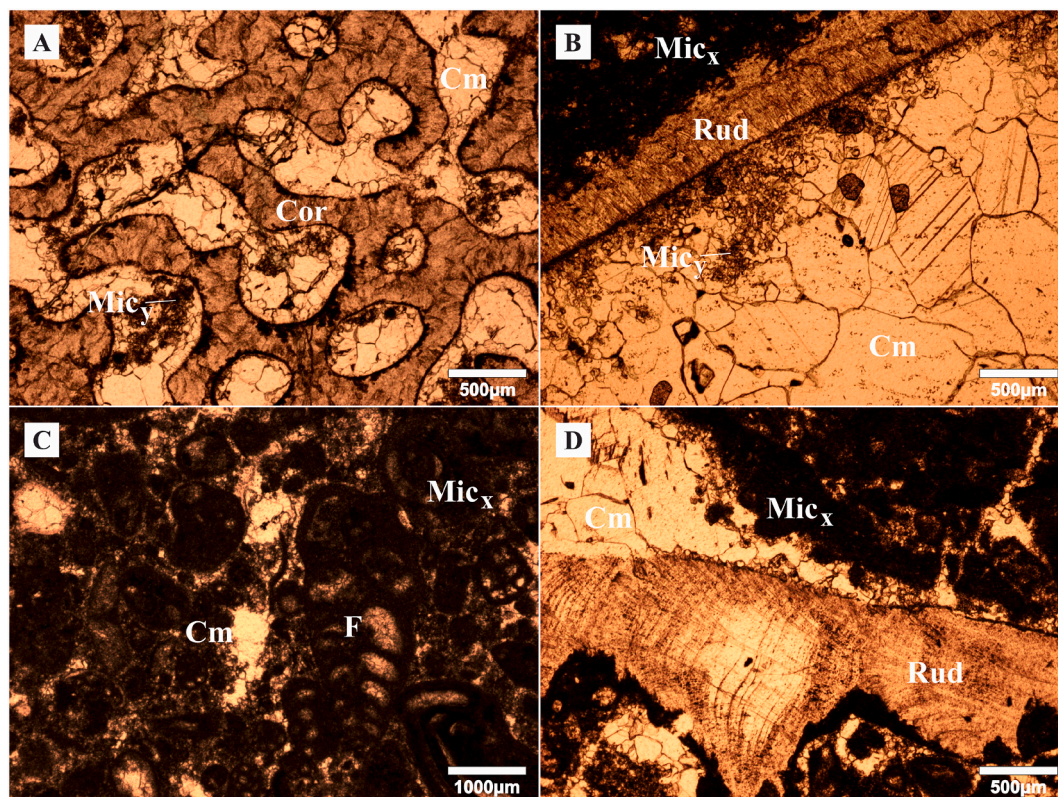


Fig. 2. Photomicrographs with plane light. A. Sample M92641 of Ibrahim-919 well, coral (Cor) with equant calcite cement (Cm) formed along the micrite (Mic_y); B. Sample X90704 of Ibrahim-973 well, rudist skeletal floatstone (Rud) with equant/blocky calcite cement (Cm); C, D: Sample L90088 of the Ibrahim-9139 well, rudist skeletal floatstone with micritized packstone matrix (Mic_x and F). The “Mic_y” micrite is found dispersed along (or within) this equant/blocky calcite cement and has a brighter luminescence compared to “Mic_x”.

(Bathurst, 1966). There is also micrite (“Mic_y”) found dispersed along (or within) this equant/blocky calcite cement (Fig. 2.A and B).

Based on photomicrographs of plane-polarized and crossed-polarized light, there are two sets of rudist: texturally preserved (Fig. 3.E) and partially altered rudist shells (Fig. 3.F). The preserved rudists are indicated by a better preservation of the original shell with a lateral aggrading prism of calcitic shell layer and homogenous crystal size (Fig. 3.E₂) that have a similar presentation to the Aptian rudist shell found in Crismina Fort, Portugal (Skelton, 2018). Meanwhile, the partially-preserved rudists exhibit a difference in the crystal mosaic that can be observed by comparing the smaller crystals (square mark) and bigger crystals (triangle mark) (Fig. 3.F₂). Similar partially-altered (micritized) rudist shells were found in Mt. Varchera, Italy (Frijia et al., 2015). In this study, we also found a fine-sized recrystallized-rudist segment (“Rud_m”) that has a similar texture to the enclosed matrix distinguished from the original rudists (“Rud”) (Fig. 4.G₁ to 4.H₂). The skeletal chamber and interparticle porosity of our samples are mainly filled with an equant-blocky calcite cements (“Cm”, Fig. 2.C and 2.D). Along with these equant calcite type of cements, a rhombic-crystal dolomite cement (Cm_d) with the brightest cathodoluminescence and void-filling characterization found in this study is also present (Fig. 4. H₂). Cathodoluminescence petrography reveals that our types of cement have a brighter luminescence compared to both skeletal fabric (rudists) and the packstone-wackestone matrix (Fig. 4.H₁ and 4.H₂). In summary, petrographic observations resulted in six recognizable diagenetic fabrics in the study area (Fig. 5): micritized-matrix, micritized bright luminescent rudist, dark luminescent rudist, bright-luminescent micrite, equant/blocky calcite cement, and dolomite cement.

4.2. Clumped isotope (Δ_{47}), $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ results

The $\delta^{18}\text{O}$ values (Table 2; Fig. 6) range from -7.87 to -5.23‰ with an average value of -7.18‰ for the matrix (Mic_x), -7.23 to -5.31‰ with an average value of -6.6‰ for the skeletal grains (Rud) and -8.98 to -8.88‰ with an average value of -8.93‰ for the blocky calcite cement (Cm). The $\delta^{18}\text{O}$ values increase proportionally to burial depth (Fig. 6.B). The $\delta^{13}\text{C}$ values range from $+2.23$ to $+3.24\text{‰}$ with an average value of $+2.85\text{‰}$ for the matrix, $+2.32$ to $+3.24\text{‰}$ with an average value of $+2.89\text{‰}$ for the skeletal grain; and $+3.48$ to $+3.51\text{‰}$ with an average value of $+3.49\text{‰}$ for the cement. There is no noticeable trend in the $\delta^{13}\text{C}$ value with depth (Fig. 6.C). The heaviest $\delta^{18}\text{O}$ and lightest $\delta^{13}\text{C}$ comprised a matrix and skeletal grain from the deepest sample in the onshore field Ibrahim-922 well. The cement, however, shows a noticeable isotopic signature difference compared to other carbonate fabrics (Fig. 7). Our $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ bulk isotopic results are consistent with previous analyses of the Barremian carbonate reservoir matrix (Vahrenkamp et al., 2014; Barata et al., 2015) and cement (Cox et al., 2010). Our data showed a narrower range of maximum and minimum values for each rock component compared to previous studies, possibly due to sampling from a narrower stratigraphic interval.

The Δ_{47} values range from 0.456 to 0.490‰ ($n = 13$) with an average value of $0.470 \pm 0.017\text{‰}$ for the matrix, 0.451 to 0.507‰ ($n = 6$) with an average value of $0.581 \pm 0.028\text{‰}$ for the skeletal grain, and 0.458 to 0.459‰ ($n = 2$) with an average value of $0.459 \pm 0.005\text{‰}$ for the cement (the laboratory’s carbonate standards uncertainty is used for the cement). The clumped isotope temperature ranges from 68 °C to 90 °C ($n = 13$) with an average value of $79.5 \pm 11\text{ °C}$ for the matrix, 60 °C to 72 °C ($n = 6$) with an average value of $68 \pm 6\text{ °C}$ for the skeletal grain, and 85 °C to 86 °C ($n = 2$) with an average value of $85.5 \pm 3\text{ °C}$ for the cement (Table 1). There was a subtle temperature (TD_{47}) decline

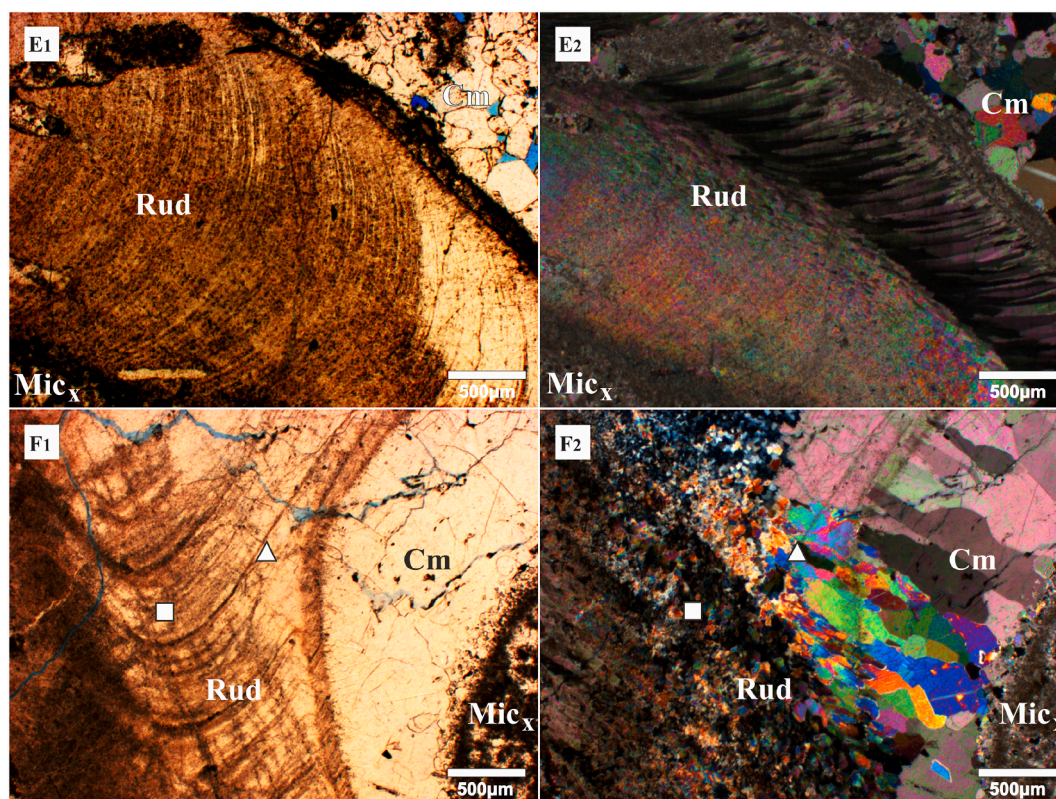


Fig. 3. Photomicrographs of rudist shells with plane light (left) and crossed-polarized light (right). E₁, E₂. Sample K89220 of the Ibrahim-911 well, with evidence of a better preserved lateral aggrading prism of the shell with homogenous calcite crystal size; F₁, F₂. Sample N97205 of the Ibrahim-922 well, with evidence of textural difference within the rudist shell of the smaller crystal size mosaic (white square) compared to the bigger crystal mosaic (white triangle) showing partially altered rudist shell.

with an increase in burial depth. The temperatures obtained on skeletal grains are also lower than the temperatures of matrix and cements at the same depth (Fig. 6.A). The clumped isotope temperatures (Fig. 5.A) are lower by 32 °C to 70 °C compared to current burial temperatures based on the regional (15 fields) geothermal gradient (Ehrenberg et al., 2020).

5. Discussion

5.1. Water-rock ratio and recrystallization of different rock fabrics

Except for the samples from the onshore Ibrahim-922 well, the matrix and skeletal grains all formed a ‘major group’ (Fig. 8, group 2). This major group is a cluster of bulk isotope ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) data from most of our samples (13 out of 17) and comprised the matrix (Mic_x), skeletal grain (Rud), and micritized skeletal grain (Rud_m) with a distinctive $\delta^{13}\text{C}$ range of +2.57 to +3.41 ‰ VPDB and $\delta^{18}\text{O}$ of −7.87 to −6.44 ‰ VPDB (Fig. 8, group 2). This major group appeared to be similar (overlap) the bulk isotope value to Field “B” with $\delta^{13}\text{C}$ range from +2.8 to +3.9 ‰ VPDB and $\delta^{18}\text{O}$ of −6.8 to −4.9 ‰ VPDB (Vahrenkamp et al., 2014). The deepest sample in the Ibrahim-922 well, which located in the flank of the anticline, collected below the oil-water contact (free-water level, 100% water saturation) of the reservoir, consists of two samples with the heaviest $\delta^{18}\text{O}$ and lightest $\delta^{13}\text{C}$ composition (Fig. 8, group 3) distinguished from the major group possibly due to different diagenetic processes. Further discussion is focused on the major group and the cement to address our first objective, which is to further understand the response of different fabrics to thermal resetting concerning clumped isotopes (Fig. 8, group 1 and 2). Previous clumped isotope temperatures for the matrix carbonates of this Barremian reservoir range from 53 °C to 84 °C (Barata et al., 2015) and 59 °C to 72 °C (Vahrenkamp et al., 2014), while temperatures derived from conventional oxygen isotopes range

from 29 °C to 40 °C (Oswald et al., 1995). Matrix temperatures recorded in this study are 68 °C to 90 °C. Based on the maximum temperature reported in all studies (Oswald et al., 1995; Vahrenkamp et al., 2014; Barata et al., 2015), solid-state reordering of clumped isotopes within minerals is unlikely as a temperature of >120 °C is required (Henkes et al., 2014). The isotopic composition and the temperature (Δ_{47}) record of our major group are therefore affected exclusively by fluid-rock interaction.

In the subsurface, fluid-rock interaction could prompt recrystallization (dissolution-(re)precipitation). The term recrystallization is used to describe a process involving dissolution and precipitation that usually involves changes in crystal size or shape without a change in mineralogy (Flügel, 2010). We use this term in our study to contrast/differentiate the cementation process where the crystallization of carbonates in the cavities does not involve simultaneous in-situ dissolution and precipitation. To gain more insight into the recrystallization events, it is crucial to also analyse the oxygen isotopic composition ($\delta^{18}\text{O}_{\text{fluid}}$) of the diagenetic fluid. This is possible by combining Δ_{47} temperature with $\delta^{18}\text{O}_{\text{carbonates}}$ to calculate $\delta^{18}\text{O}_{\text{fluid}}$ using the (Kim and O’Neil, 1997) equation (Table 2 and Fig. 9). In low water-rock ratio conditions, isotope-exchange reactions occur in a rock-buffered system where $\delta^{18}\text{O}$ compositions of the recrystallized minerals may not change through time (Sheppard, 1986; Shenton et al., 2015; Huntington and Lechler, 2015). In contrast, the mineral’s $\delta^{18}\text{O}$ in a high water-rock ratio will be transformed/enriched, while the $\delta^{18}\text{O}$ of the diagenetic fluid will tend to be invariable (Sheppard, 1986; Shenton et al., 2015; Huntington and Lechler, 2015).

5.1.1. Water-rock ratio and diagenetic fluid of cement and skeletal grain

Except for the matrix, the calculated $\delta^{18}\text{O}_{\text{fluid}}$ values are similar to those reported in previous studies, +0.89 to +4.13 ‰ SMOW (Barata

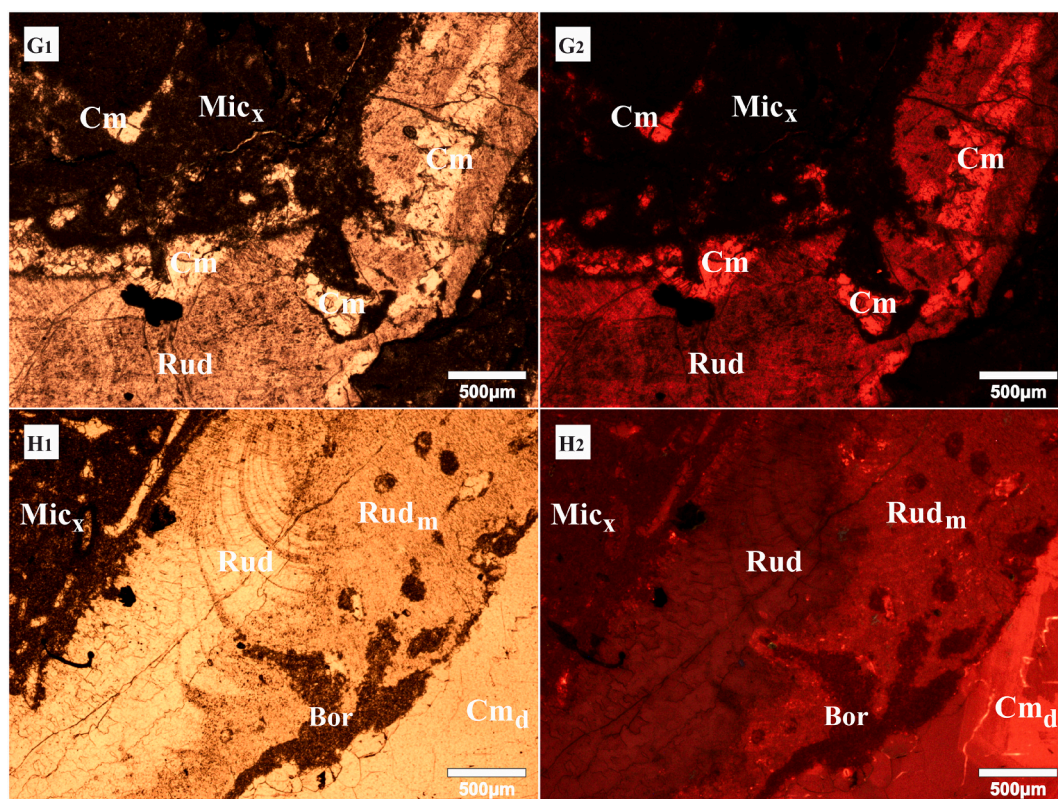


Fig. 4. Photomicrographs with plane light (left) and cathodoluminescence (right). G1, G2. Sample M92641 of the Ibrahim-919 well from the onshore field, with evidence of equant/blocky calcite cements replacing the mud matrix and plugging most of the intraparticle porosity (Cm); H1, H2. Sample Z94708 of the Rainy-9316 well from the offshore field, with evidence that (1) calcite cements formed within interparticle porosity, (2) rudists are partially recrystallized (Rud_m), and (3) there are occurrences of dolomite cement (Cm_d). The skeletal fabric shows a dissolution pattern interpreted here as a micritization remnant related to seafloor diagenesis.

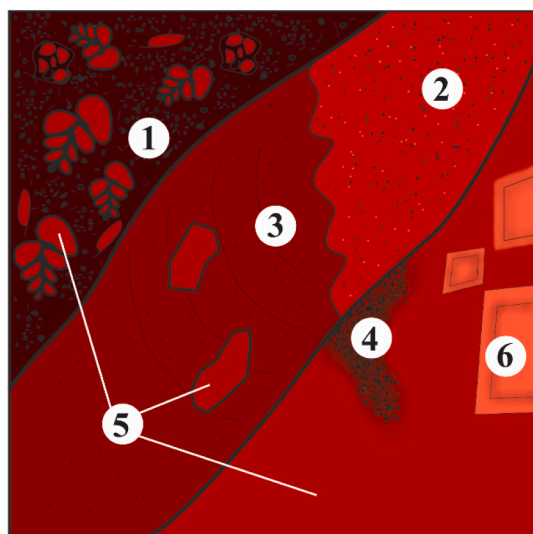


Fig. 5. Carbonate diagenesis model within the Barremian carbonate reservoir zone in this study, based on both polarization and CL photomicrograph. Chronological events include: Early seafloor micritization processes of (1) micritized-matrix “Mic_x” and (2) micritized-bright luminescent rudist “Rud_m”; Recrystallization processes associated with burial diagenesis of (3) dark luminescent rudist “Rud” and (4) bright-luminescent micrite “Mic_y”; Burial-cementation processes of (5) equant/blocky calcite cement “Cm” and (6) dolomite cement “Cm_d”.

et al., 2015) and +3.3 to +4.9‰ SMOW (Vahrenkamp et al., 2014). A plot of the calculated $\delta^{18}\text{O}_{\text{fluid}}$ (SMOW) against $\text{T}\Delta_{47}$ and the $\delta^{18}\text{O}$ calcite isolines (Fig. 9) shows that the cements are formed at 85 °C with a $\delta^{18}\text{O}_{\text{fluid}}$ of 3.8 ‰ SMOW, which is within error of the skeletal grain $\delta^{18}\text{O}_{\text{fluid}}$ values (3.07–4.33‰, Fig. 9). The diagenetic fluid ($\delta^{18}\text{O}_{\text{fluid}}$) consistency between the cement and the skeletal grains (3.07–4.33‰), compared to the enrichment of its mineral’s $\delta^{18}\text{O}_{\text{Ca}}$ composition (−9.0 to −5.3‰), confirms the large water-rock ratios during recrystallization processes in our formation. This large water-rock ratio environment is predictable, as it was also isotopically recognized in a diagenetic fluid study of the Arabian Gulf Coast (Sheppard, 1986) and Palmarito Formation, Venezuela (Shenton et al., 2015).

The skeletal grain (Rud) comprised a bigger crystal size compared to enclosed micrite (i.e. Fig. 2.D and Fig. 4.H) with lower temperature ($\text{T}\Delta_{47}$). The lowest recorded temperature of skeletal grain (60 °C, 1SE = 6 °C) retains the closest temperature to the 32 °C to 43 °C corrected-sea surface temperature of Barremian to Aptian period at $\pm 30^\circ$ paleo-latitude (O’Brien et al., 2017). Therefore, we suggest that this fabric retains the temperature of the early burial stage, which is closer to the temperature record during deposition, possibly because the bigger grain size prevents later episodes of recrystallization. The smaller crystal of the micritized-skeletal segment (Rud_m), however, has experienced a recrystallization process in a low water-rock ratio based on the similarity of the isotopic characteristics ($\delta^{18}\text{O}_{\text{fluid}}$ and $\delta^{18}\text{O}_{\text{calcite}}$) to the enclosed matrix (Fig. 9). This micritized-skeletal segment records a temperature of 90 °C (1SE = 6 °C) closer to the present-day formation temperature (122 °C, 2746 m), and a temperature at the upper end of the enclosed matrix (68 °C to 90 °C; $n = 13$) (Fig. 9).

5.1.2. Water-rock ratio and diagenetic fluid of the matrix

A plot of the calculated $\delta^{18}\text{O}_{\text{fluid}}$ (SMOW) against $\text{T}\Delta_{47}$ and the $\delta^{18}\text{O}$

Table 2

Carbonate clumped isotope values (Δ_{47}) and bulk isotope composition ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) of reservoir samples at both study locations. The fluid $\delta^{18}\text{O}$ SMOW were calculated with the Kim and O'Neil (1997) equation. The skeletal fragment/rudist is indicated with an 'sk' extension on the end of the sample number. The matrix samples are indicated by 'm1' and 'm2', and the cement by 'cem' extensions after the sample number. Sample Y90101_sk is a micritized rudist and is treated as a matrix.

Sample ID	Depth (m)	n	$\delta^{13}\text{C}$ (‰, VPDB)	$\delta^{13}\text{C}$ SE (‰)	$\delta^{18}\text{O}$ (‰, VPDB)	$\delta^{18}\text{O}$ SE (‰)	$\delta^{18}\text{O}$ fl. ^a (‰, SMOW)	$\delta^{18}\text{O}$ fl. SE (‰)	Δ_{47} (‰, I-CDES)	Δ_{47} SE (‰)	Δ_{47} Temperature ^b (°C)	Δ_{47} T. SE (°C)
<i>Onshore Giant Field</i>												
K89220_m	2719	4	2.57	0.02	-7.47	0.04	4.95	0.32	0.463	0.004	82	2
K89220_sk	2719	3	2.57	0.03	-7.14	0.11	3.76	0.38	0.482	0.006	72	3
L90004_m	2743	3	2.82	0.02	-7.29	0.10	4.68	0.32	0.469	0.005	79	3
L90088_m	2745	3	3.41	0	-7.69	0.05	5.27	1.15	0.457	0.014	86	9
L90088_sk	2745	3	2.84	0.01	-6.82	0.05	4.09	1.16	0.482	0.016	72	9
L90088_cem	2745	3	3.51	0.01	-8.98	0.09	3.86	0.81	0.458	0.011	86	7
M92641_m	2823	3	3.18	0.01	-6.73	0.03	4.18	0.09	0.482	0.001	72	1
M92836_sk	2829	4	3.18	0.02	-7.23	0.03	3.07	0.84	0.490	0.011	68	6
N97205-m ^(c)	2963	3	2.25	0.02	-5.23	0.14	5.13	0.69	0.490	0.011	68	6
N97205-sk ^(c)	2963	3	2.23	0.03	-5.31	0.08	3.73	0.91	0.507	0.013	60	6
W957510-m ^(c)	2918	3	3.31	0	-7.26	0.04	4.10	1.05	0.476	0.014	75	8
W957510-sk ^(c)	2918	3	3.24	0.03	-6.44	0.04	4.33	0.98	0.484	0.013	71	7
X90571_m	2760	3	2.86	0.02	-7.01	0.03	4.12	0.82	0.479	0.011	74	6
X90704_m2	2764	3	3.41	0.04	-7.84	0.12	5.20	0.75	0.456	0.009	87	5
X90704_m1	2764	3	3.27	0.05	-7.8	0.03	4.77	0.25	0.461	0.003	84	2
X90704_cem	2764	3	3.48	0.05	-8.88	0.12	3.82	0.99	0.459	0.012	85	7
Y90101_m2	2746	4	3.29	0.03	-7.21	0.05	5.38	0.83	0.461	0.011	84	7
Y90101_m1	2746	4	2.88	0.03	-6.98	0.04	4.76	0.09	0.472	0.001	78	1
Y90101_sk (mic)	2746	4	3.11	0.02	-7.87	0.09	5.56	0.81	0.451	0.010	90	6
<i>Offshore Giant Field</i>												
Z94708_m	2886	3	3.14	0.09	-7.04	0.18	4.17	0.86	0.478	0.012	74	7
Z94708_sk	2886	3	3.09	0.10	-6.83	0.05	3.11	0.56	0.494	0.007	66	4

^a Calculated diagenetic fluid $\delta^{18}\text{O}$ SMOW from calcite $\delta^{18}\text{O}$ using Kim and O'Neil (1997) equation.

^b The calculated subsurface temperature from Δ_{47} value using Anderson et al. (2021) equation.

^c Samples obtained below oil water level.

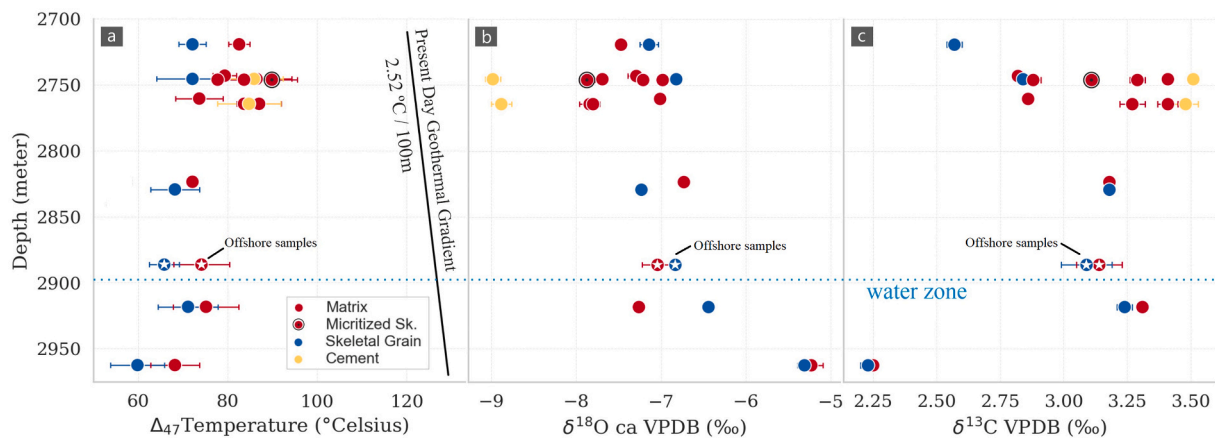


Fig. 6. Results of (a) Δ_{47} (b) $\delta^{18}\text{O}$ and (c) $\delta^{13}\text{C}$, all plotted against depth for both study locations. The present-day geothermal gradient in the Barremian carbonate reservoir temperature obtained from a previous study of 15 fields in onshore and offshore areas (Ehrenberg et al., 2020) is also shown. The water zone is located at the base of a hydrocarbon column and the transition zone of the reservoir, where the water saturation (S_w) is 100%. The offshore field data at 2886 m depth is marked by a star symbol.

calcite isolines (Fig. 9) shows that the $\delta^{18}\text{O}_{\text{calcite}}$ of the matrix is near constant at -7% and clumped isotope temperatures vary broadly ($66\text{--}90\text{ }^{\circ}\text{C}$) resulting in a large spread in calculated $\delta^{18}\text{O}_{\text{fluid}}$ (Fig. 9, Trend 2). This shows that the $\delta^{18}\text{O}$ enrichment in the diagenetic fluid related to the matrix is similar to the rock-buffered system or 'closed-system pathway' in the Palmarito Formation, Venezuela and the Bird Spring Formation, Nevada (Shenton et al., 2015; Huntington and Lechler, 2015). All of these conditions support (Budd, 1989)'s previous study that suggested a low water-rock diagenetic environment in non-porous rocks (i.e., matrix), whereas other rock textures in the same

formation (in this case, rudist wackestone/packstone) show evidence of a higher water/rock ratio. However, our findings suggest that a crystal size could have more impact to the mineral's proclivity towards recrystallization exceeding the water/rock ratio aspect, as the larger crystal size (large skeletal grain) tend to recrystallize early with lower temperature in contrast to the more finely crystalline (enclosed matrix) which tend to be more reactive and continue to record a wide range of temperature towards the maximum burial condition. Thus, we also suggest that the matrix (micrite) is a more useful recorder of maximum burial conditions and can be key to reconstructing a detailed thermal

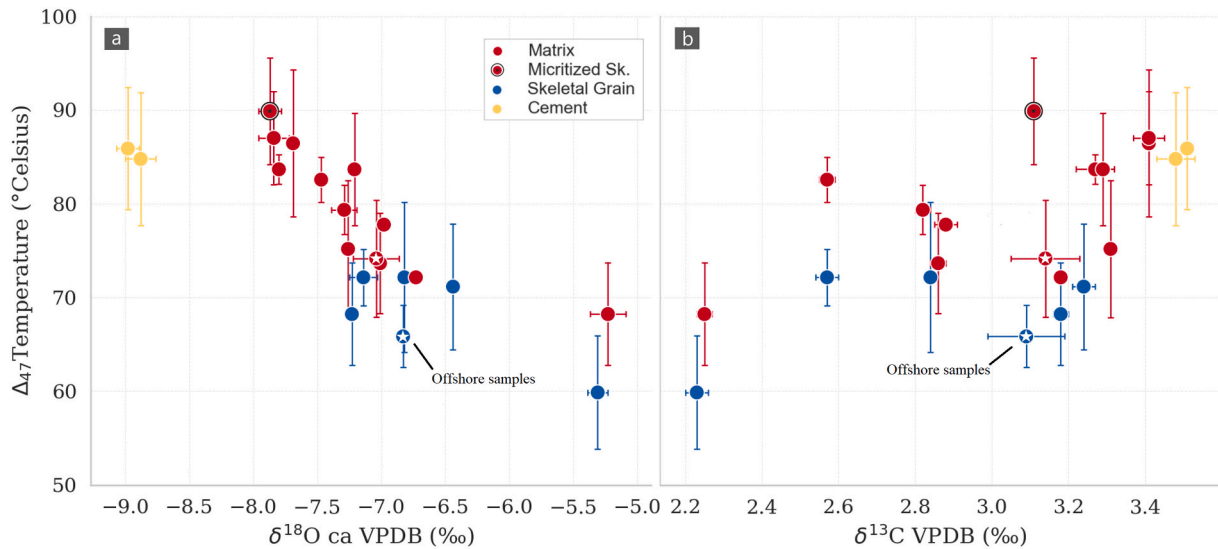


Fig. 7. Results of (a) $\delta^{18}\text{O}$ and (b) $\delta^{13}\text{C}$ with respect to Δ_{47} -temperature of reservoir's matrix, skeletal grain, and cement. The Δ_{47} -temperature was calculated using the Anderson et al. (2021) equation. No distinctive isotopic difference between the onshore and offshore samples.

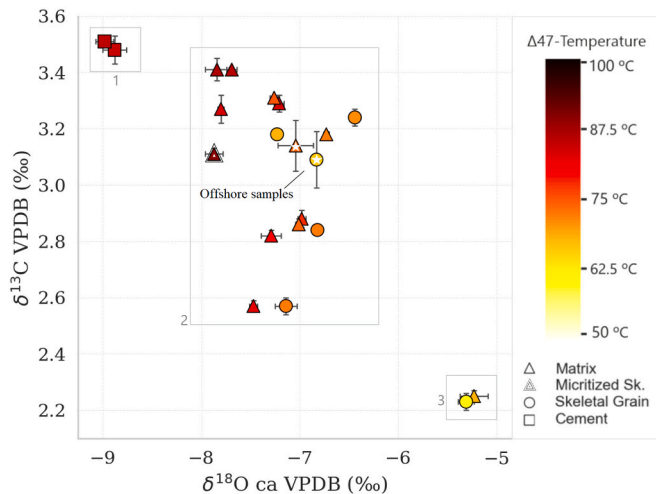


Fig. 8. The combination of $\delta^{18}\text{O}$, $\delta^{13}\text{C}$ and Δ_{47} -temperature relationship suggests that there are three distinctive isotope groups. Group 1 comprises only cements, group 2 comprises a matrix, skeletal grain and micritized skeletal grain with a range of recorded temperatures. The offshore samples (star symbol) fit into group 2. Group 3 comprises a matrix and skeletal grain from the Ibrahim-926 well (N97205).

and diagenetic history.

5.2. Timing of recrystallization for each fabric

Attempts to studying the relationship between oil emplacement and diagenesis have been performed in this reservoir using oil-fluid inclusion and porosity trend analysis (Neilson et al., 1996; Neilson et al., 1998). Here, we used the established calibrated-burial and paleotemperature model of the reservoir (Barata et al., 2015), to connect the broad recrystallization-temperature record from our clumped isotope result with this model (Barata et al., 2015) and track the recrystallization age for our carbonate fabrics based on the recorded temperature. Using this approach, a composite burial history graph was created (Fig. 10). The Barremian carbonate reservoir burial is shown by black burial-history lines in Fig. 10. The TA_{47} of our results are categorized by each fabric and shown as a matrix (red), micritized matrix (red with dark circle),

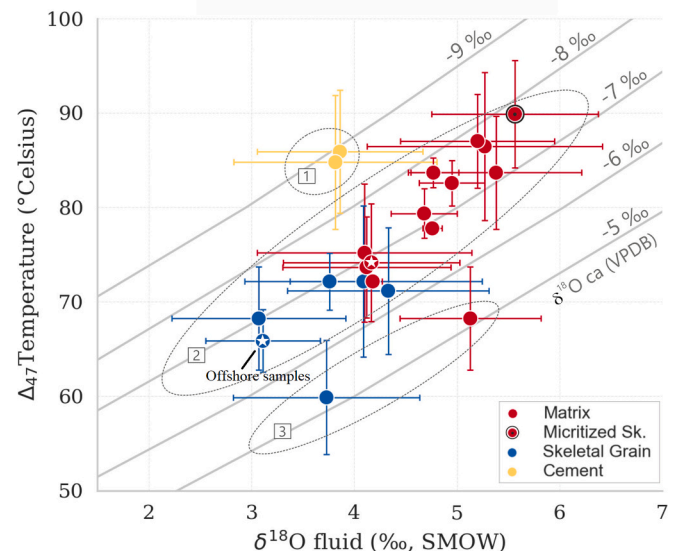


Fig. 9. Calculated Δ_{47} -temperature versus the calculated $\delta^{18}\text{O}$ of the diagenetic fluid.

Values were reconstructed using the Kim and O'Neil (1977) equation. Both skeletal fragment and cement appear to have a lower $\delta^{18}\text{O}_{\text{fluid}}$ compared to the matrix. The offshore samples (star symbol) are indistinguishable from the onshore samples. The main pattern of the three-isotopic group from the previous plot (Fig. 8, $\delta^{18}\text{O}$ vs $\delta^{13}\text{C}$) can still be recognized. The data distribution along the -7‰ (VPDB) $\delta^{18}\text{O}_{\text{ca}}$ line seems to show precipitation along a constant $\delta^{18}\text{O}$ calcite line with increasing temperature (group 2).

skeletal grain (blue), and cement (yellow). The data's 95% confidence level is translated into an age error and plotted in the same coloured line.

Skeletal grains are likely to have recrystallized during shallow (0.9–1.3 km) burial given the fact that they record temperatures between 60 °C and 72 °C. Using the existing temperature and time of the calibrated-burial history model in the area (Barata et al., 2015), the timing of precipitation (recrystallization) of the skeletal grains can be traced and determined between 98 and 70 Ma (Blue marker, Fig. 10). Note that the uncertainties aspect of this burial history is not explained in the previous report, and thus cannot be accounted for in our study. Compared to the skeletal grains, the cements are precipitated at a

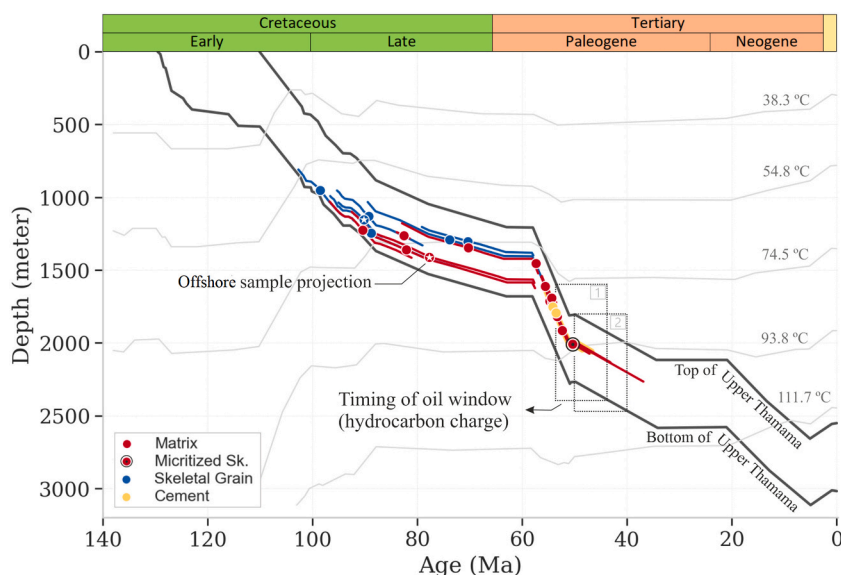


Fig. 10. Burial history of Barremian carbonate formation from the onshore field by Barata et al. (2015) and Ehrenberg et al. (2020a). The dashed box represents the report of hydrocarbon charge timing. Box number 1, the oil window and hydrocarbon charging reported at 40–50 Ma (Taher, 1996); box number 2, the hydrocarbon charge reported at 54–55 Ma in the onshore and 44 Ma in the offshore area (Gumati, 1993). The temperatures recorded by clumped isotopes (coloured symbols) indicate the stopping of recrystallization at a time compatible with oil emplacement. The offshore samples are projected to this burial history based on similarities between temperature-depth relationships (Ehrenberg et al., 2020a).

greater burial depth and higher burial temperatures (85 °C to 86 °C), implying a timing of precipitation between 54 and 53 Ma (Yellow marker, Fig. 10). The timing of cement precipitation is compatible with Cenozoic-tectonic events that led to rapid burial (Fig. 10) followed by uplift and tilting towards the NE along with thrusting and folding in the east of Abu Dhabi.

Meanwhile, the matrix (Red marker, Fig. 10) seems to continuously recrystallize and record a wide range of temperatures between 68 and 90 °C. This implies a timing for their continuous recrystallization between 90.4 and 50.4 Ma (Red marker, Fig. 10). Our result is at odds with a previous diagenesis study of this formation's matrix, which reported that it recrystallizes earlier in the Cenomanian to Santonian 100–83 Ma (Barata et al., 2015) or Turonian-Campanian 93–72 Ma (Vahrenkamp et al., 2014). We note that a major difference between our study and the two previous studies (Barata et al., 2015; Vahrenkamp et al., 2014) is that we have analysed separate diagenetic fabrics, whereas the previous authors analysed bulk-rock. We argue that our multi-phase clumped isotope record thus sheds light on the detailed diagenetic history of this formation.

5.3. Oil emplacement and diagenesis stoppage

The observation that matrix temperatures are 32 °C to 53 °C lower than present-day reservoir temperatures in the two fields indicates that the matrix continuously recrystallized through the early burial but ceased reacting before reaching the current maximum burial temperature. Notice that the same process of dissolution-(re)precipitation cessation also occurred below the current oil zone. This condition is likely due to a hydrocarbon adherence on the carbonate surfaces (preferentially oil-wet, wettability) (Zhang and Austad, 2006; Sagbana et al., 2022) which diminish its chemical reactivity even when the pores are filled with water (Frye and Thomas, 1993). The last recrystallization temperature recorded in our samples is 90 °C, which can be translated into the timing of the last recrystallization at 50 Ma. The exact age is difficult to pin exactly, and using the 95% confidence interval results in an estimated range of 53 to 37 Ma (Red line extension, Fig. 10).

Evidence for oil migration events and the timing of hydrocarbon emplacement in the cement of the reservoir has been previously studied using petroleum-fluid inclusion (Gumati, 1993; Neilson et al., 1996; Taher, 1996; Neilson et al., 1998). The best estimate for the timing of hydrocarbon charging in our carbonate formation is reported between 40 and 50 Ma (Taher, 1996) (Dashed box number 2, Fig. 10), whereas another study suggested 54–55 Ma for the onshore reservoirs and 44 Ma

for the offshore area (Gumati, 1993) (Dashed box number 1, Fig. 10). The strong overlap between the documented ages for hydrocarbon emplacement and the timing of the last recrystallization event suggests that the end of carbonate diagenesis in this reservoir is synchronous with hydrocarbon charging (Fig. 10). Thus, it is likely that the inhibition of diagenesis in our zone was a consequence of the displacement of water by hydrocarbons.

Whether diagenesis (i.e., cementation) continues after hydrocarbon emplacement has been debated for years (Feazel and Schatzinger, 1985; Scholle and Halley, 1989; Walderhaug, 1990; Ramm, 1992; Oelkers et al., 1992; Neilson et al., 1996; Neilson et al., 1998; Cox et al., 2010; Kolchugin et al., 2016). Our results do not support the idea that cementation occurred after petroleum charging (Burruss et al., 1985) or that hydrocarbon charging occurred in the Late Cretaceous for these reservoirs (Alsharhan, 1989; Oswald et al., 1995). Instead, our study supports models that suggest that oil displacement of pore water in the Cenozoic led to the retardation of cementation (Neilson et al., 1996; Neilson et al., 1998). The exact mechanisms responsible for this process are beyond the scope of this study, but previous work has suggested that for carbonate minerals the inhibition of new cement nucleation of carbonate already begins at lower oil saturation in the pore space (Cox et al., 2010), and that selective loss of permeability occurred because of the closing of pores due to hydrocarbons (Cox et al., 2010).

Beyond lending support to the idea of hydrocarbon migration limiting carbonate diagenesis, our results also highlight the potential applications of clumped isotopes in petroleum system analysis.

6. Conclusion

We demonstrate that different carbonate fabrics recrystallize at different water/rock ratios and that the finer-grained matrix continued to recrystallize even at a low water/rock ratio during the deeper burial history in reservoir rocks in the studied Barremian limestone interval. The matrix records a wide range of temperatures, suggesting continuous recrystallization between 90 and 50 Ma based on established thermal histories. The maximum temperature recorded by the matrix is lower than the present-day formation temperature, suggesting the stoppage of dissolution-(re)precipitation diagenetic reactions before the time of maximum burial.

Based on a composite burial history model, this diagenesis stoppage coincides with the timing of oil emplacement. Whether oil emplacement retards diagenesis (cementation) has been debated, and our findings potentially provide a conclusive element for this debate in carbonate

systems. Our results also extend the application of clumped isotope analyses to petroleum migration studies.

Declaration of Competing Interest

The authors declare that they have no known financial interests or personal relationships that could have influenced the work reported in this paper.

Data availability

Data will be made available on request.

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