

Article

Clumped Isotope Reordering and Kinetic Differences in Co-Hosted Calcite and Dolomite Minerals throughout Burial Diagenesis and Exhumation

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Abstract: The clumped isotope paleo-thermometer has become a valuable proxy for the burial history reconstruction of carbonate formations. To maximise the accuracy of these reconstructions, post-depositional alterations, such as recrystallisation and Δ_{47} isotope exchange reactions, must be understood. In this study, we examine the isotopic behaviour of calcites and early dolomite samples from the same stratigraphic intervals, and thus with similar burial history. This approach provides additional constraints on the kinetics of Δ_{47} reordering in dolomite during exhumation. Clumped isotope measurements were performed on 19 calcites and 15 early dolomites from the Permian, Jurassic, and Cretaceous periods from four locations in Oman spanning different burial regimes. The calcite and dolomite samples were collected from the rock matrix, based on the assumption that fine material was more susceptible to recrystallisation. Our results show that calcites and dolomites record different Δ_{47} values despite being subjected to the same thermal history. The maximum Δ_{47} temperature recorded in dolomites (181 ± 13 °C) corresponds to the oldest and most deeply buried Permian rock. This value is approximately 35 °C higher than those measured in the co-located and coeval calcite matrix (145 ± 14 °C). This discrepancy suggests that calcite and dolomite have different kinetic parameters. Our data confirm (1) that dolomite Δ_{47} values are more resistant to alteration during burial and exhumation than Δ_{47} calcite values, and (2) that dolomite has a higher Δ_{47} closing temperature than calcite during cooling. The presence of two mineral phases with distinct kinetic parameters in the same stratigraphic unit provides additional constraints on models of burial and uplift. In addition, mineralogical data coupled with Δ_{47} and burial depths suggest that the progressive development of dolomite cation ordering is driven by temperature elevation, as previously suggested.

Keywords: clumped isotopes; burial history; solid-state reordering; dolomite cation ordering



Citation: Adlan, Q.; Kaczmarek, S.E.; John, C.M. Clumped Isotope Reordering and Kinetic Differences in Co-Hosted Calcite and Dolomite Minerals throughout Burial Diagenesis and Exhumation. *Minerals* **2023**, *13*, 1466. <https://doi.org/10.3390/min13121466>

Academic Editors: Martino Giorgioni, Luca Basilone, Tomaso Bontognali and Luigi Jovane

Received: 25 August 2023

Revised: 1 November 2023

Accepted: 17 November 2023

Published: 22 November 2023



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1. Introduction

Clumped isotope (Δ_{47}) thermometry is a technique that ideally recovers the temperature of the formation or recrystallisation ($T_{\Delta_{47}}$) of all carbonate phases without relying on knowledge of the $\delta^{18}\text{O}$ value of the formational fluid [1,2]. This technique has been widely employed to reconstruct the burial history of carbonate strata in sedimentary basins [3–19]. Although the study of clumped isotopes in buried carbonates offers great promise in resolving many geological problems, concerns about post-depositional alteration of the Δ_{47} values, as a result of the effects of burial and exhumation, including diagenetic modification via dissolution–precipitation recrystallisation, have emerged, particularly in rock-buffered (i.e., closed) systems [7,8,11,18,20,21].

In a rock-buffered system, the recrystallisation process occurs at low water–rock ratio conditions along with the associated Δ_{47} isotope exchange reactions. In this case, the $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ values remain invariant (or incur minor variations) while the Δ_{47} values reflect the ambient burial or exhumation temperature. Even if no recrystallisation occurs, the Δ_{47} values may be reset during a process known as solid-state reordering [7,8,11,18,20,21]. Solid-state reordering during the exhumation process can drive the Δ_{47} values to record the apparent equilibrium or blocking temperature of the mineral [7,8,22], which is related to the cooling rate of the strata [8,23]. This blocking temperature is independent of the original crystallisation temperature and peak burial temperature [7,22]. Understanding the solid-state reordering process and its effects on Δ_{47} values could provide details of the thermal and diagenetic histories of carbonate formations during maximum burial through exhumation.

Investigations of solid-state reordering in different carbonate minerals have been performed to help constrain burial–exhumation histories. Using controlled heating experiments, kinetic parameters have been evaluated in calcite [7,8,20,24], dolomite [21], and aragonite [25,26]. The cooling rates from these experiments have been inferred from as much as ten degrees Celsius per day to as little as a few degrees Celsius per million years [8]. These kinetic parameters have been used as inputs in models to predict the change in Δ_{47} in response to different thermal scenarios. To date, such models have been used to constrain the temperature–time (T–t) histories during burial and exhumation in sedimentary basins [10,11,13,15,27–29] and metamorphic environments [16,22].

Carbonate minerals, such as calcite and dolomite, are common in the sedimentary rock record. Of the two, calcite is more commonly used to reconstruct burial history [9,13,29]. The sample selection of dolomite needs to be carried out carefully, as hydrothermal dolomite replacement can sometimes happen under the influence of high-temperature fault-controlled hydrothermal fluids [30–32]. Meanwhile, different carbonate rock fabrics in a single hand specimen can record different recrystallisation temperatures reflecting different stages of burial [33]. For example, it has been suggested that fine-grain carbonates are more susceptible to recrystallisation during diagenesis [12,27] and record a wider Δ_{47} temperature range [33] than their coarse-grained counterparts. These examples illustrate the complexity of Δ_{47} values in carbonate rocks, with conditions that are difficult to replicate in the laboratory. One approach to understanding the Δ_{47} exchange reactions through burial–exhumation complexity is to use geological samples as a natural laboratory, which provides a testing ground to examine the isotopic behaviour of multiple minerals that share the same burial history.

In this study, we investigate ancient carbonates across a regional orogenic belt. We used Cretaceous, Jurassic, and Permian carbonate formations deposited on a stable carbonate platform in the Arabian Peninsula to examine Δ_{47} variations in calcites and dolomites that experienced various degrees of burial and exhumation. Four locations were selected based on the inferred maximum burial depth. From shallow to deep, these include the Hausshi-Huqf in Oman, an offshore location in the Arabian Gulf, the Musandam Peninsula (UAE), and the Jabal Akhdar in the Central Oman Mountains. The objectives of this study are as follows: (1) to compare and contrast the clumped isotope records of recrystallised (early) calcites and (early) dolomites that have undergone similar burial and exhumation histories to explore potential differences in clumped isotope kinetics; (2) to investigate the cooling phase and compare the apparent blocking temperatures of calcite and early dolomite in various burial–exhumation histories; and (3) to investigate the link between cation ordering in dolomite and apparent burial temperature.

2. Geological Settings of the Locations

The stratigraphy of carbonate rocks within the Oman area provides an ideal setting to explore the clumped isotope variability of recrystallised carbonates because the same stratigraphic intervals buried to different depths can be sampled at the surface today (Figure 1) [9,34–48].

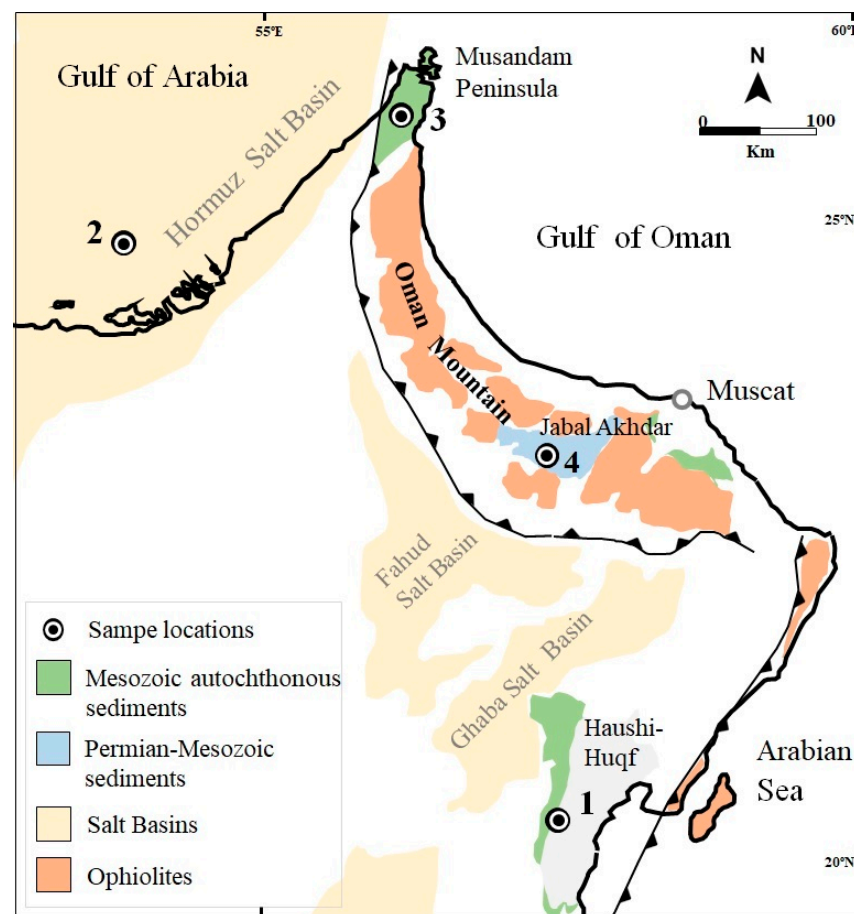


Figure 1. Study locations from shallow to deep burial tectonic settings: (1) the Haushi-Huqf in Oman; (2) offshore Arabian Gulf; (3) the Musandam Peninsula; and (4) the Jabal Akhdar, Central Oman mountains. Regional map modified from Loosveld et al. [49] and Cooper et al. [50].

The Cretaceous carbonate interval in Haushi-Huqf (Area 1, Figure 1) was deposited on a relatively stable and slow subsidence shelf [46,51] in a relatively low-temperature regime [9]. The maximum burial depth of the Cretaceous interval on the Haushi-Huqf High, based on post-Cretaceous sediment coverage (erosion surface analysis), is estimated to be 400 m [40,51], while clumped isotope measurement on recrystallised oysters suggests deeper burial depths of 1.0–1.2 km [9]. The Qishn Formation in the same area was deposited during the Barremian–Aptian stages [52] and is equivalent to the Upper Kharaib and Lower Shu’aiba formations in the northern part of Oman (Figure 2).

The Cretaceous Thamama Group, offshore Arabian Gulf (Area 2, Figure 1), comprises carbonate units that range in thickness from 685 to 762 m and can be divided into four formations: Habshan, Lekhwair, Kharaib, and Shu’aiba [53] (Figure 2). The Cretaceous interval in this area was buried to ~2500 to 3000 m, where it currently resides [54,55].

The Musandam Group is a ~1500 m thick Jurassic carbonate interval in the Musandam Peninsula (Area 3, Figures 1 and 2). This interval was overridden during the Late Cretaceous obduction by multiple thrust sheets consisting of the Hawasina complex [38,56] and ophiolites [38,57]. The burial history model was calibrated with a mixed layers illite-smectite paleothermometer and U-Pb dating suggests that the maximum burial depth of the Jurassic interval is ~5100 m [38]. Later, Eocene to Miocene uplift and erosion were followed by Neogene cooling [38,39].

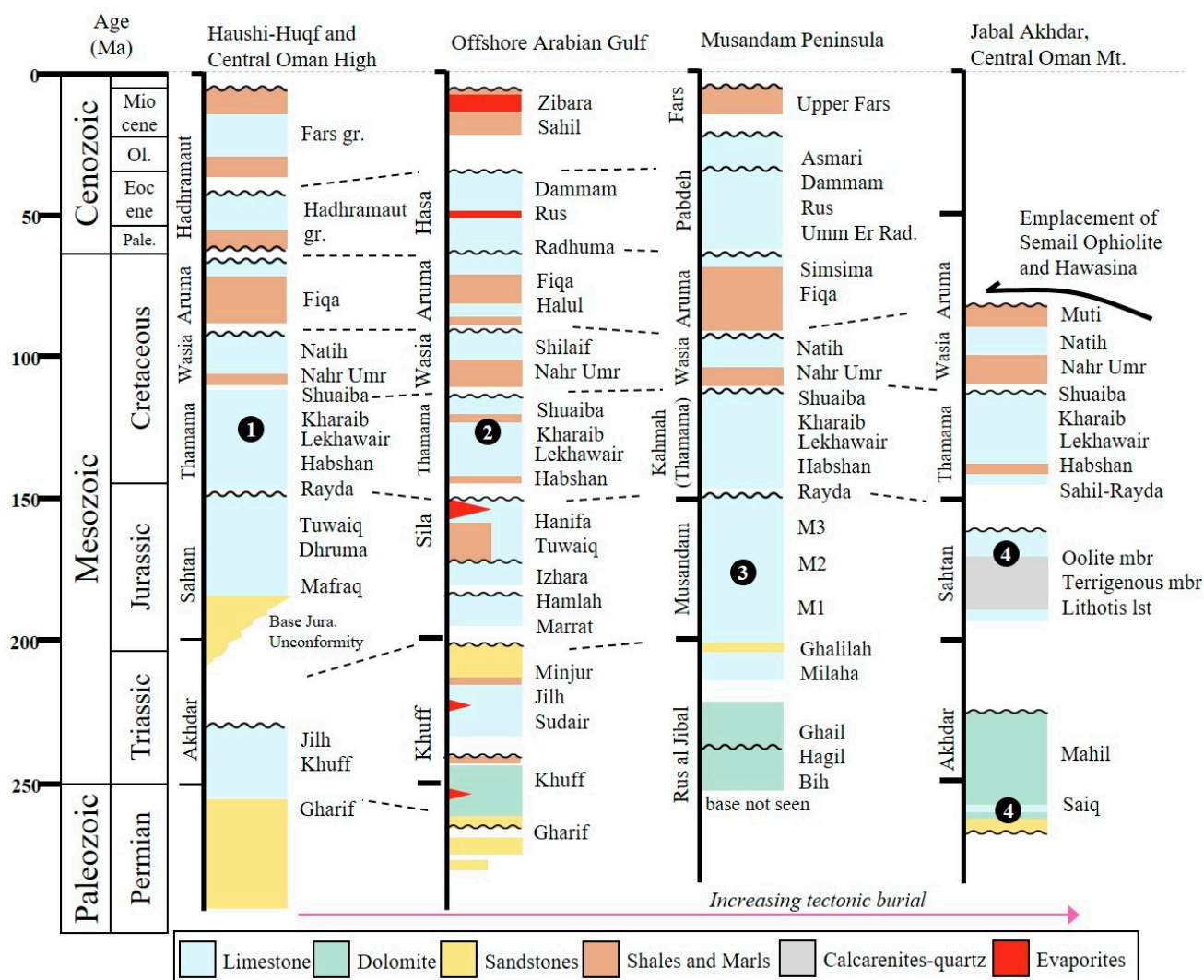


Figure 2. The sample position on the stratigraphic column of each region was modified from Loosveld et al. [49] and Cooper et al. [50]. (1) The Cretaceous Thamama Group in the Haushi-Huqf; (2) the Cretaceous Thamama Group in the offshore Arabian Gulf; (3) the Jurassic Mudandam Group in the Musandam Peninsula; and (4) the Jurassic Sahtan Group and Permian Akhdar Group on the Jabal Akhdar, Central Oman Mountains. The wavy line on top of the formation represents the unconformity/sequence boundary, and the dashed line reflects the correlation to the other adjacent region.

The Permian carbonate interval of the Akhdar Group in Jabal Akhdar, Central Oman Mountains (Area 4, Figure 1) consists of a thick succession of carbonates, including the early diagenetic dolomite (Khuff equivalent) that formed as a result of hypersaline brine reflux [36]. Meanwhile, the Middle to Upper Jurassic Sahtan Group in this area is composed of ~30 to 400 m thick carbonate units [42]. Both the Permian and the Jurassic strata in this area are overlain by allochthonous units of the Hawasina complex [38,56] and ophiolites [38,57]. The burial history model calibrated using a fluid-inclusion paleothermometer, zircon (U-Th)/He thermochronometer, and solid bitumen reflectance, shows that these strata were buried under 8–10 km with an additional 2 km of sedimentary nappes [39]. The area experienced uplift [58], folding [45], and erosion from the Miocene period to recent times, exposing the oldest units (Precambrian) in the centre, followed by younger sedimentary beds (up to the Cretaceous age) towards the outer rim [36].

3. Materials and Methods

3.1. Materials

In total, 34 samples (19 calcites and 15 dolomites) were collected in Oman and the United Arab Emirates during previous field campaigns [36,49,59,60] (Table 1).

Table 1. Location and sample source.

Area	Formation or Group	Age of Interval	Maximum Burial Depth ^(a) (m)	Total Sample	Temp. Estimation ^(b) (°C)	Specimen
1. Haushi-Huqf, Oman	Qishn Fm.	Cretaceous	400	3	47	Outcrop
2. Offshore Arabian Gulf	Kharaib Fm.	Cretaceous	2943–2950	8	130	Core
3. Wadi Naqab, Musandam Peninsula	Musandam II	Jurassic	5100	7	168	Outcrop
4. Jabal Akhdar, Central Oman Mt.	Sahtan Gr.	Jurassic	8000	6	169	Outcrop
4. Jabal Akhdar, Central Oman Mt.	Saiq Fm.	Permian	9000	16	187	Outcrop

^(a) The maximum burial stratigraphic depth of Jabal Akhdar Central Oman Mountains obtained from Grobe et al. [39]; the distance between the Jurassic and Permian sampling locations is ~1000 m; the maximum burial depth of the Musandam Peninsula was obtained from Carminati et al. [38]; Haushi-Huqf burial from Immenhauser et al. [40] and Sattler et al. [50]. ^(b) Expected temperature of the samples on maximum burial depth: the geothermal gradient for Haushi-Huqf were obtained from Bergmann et al. [37]; the offshore-Arabian Gulf from Ehrenberg et al. [55]; Musandam Peninsula from Tibat-1ST well in Ali et al. [61]; and Oman Mountain from NB-22 well in Schütz et al. [62].

Samples from the Cretaceous units in the Haushi-Huqf were laminated mudstone to wackestone limestones, and mud-dominated dolomites in the Qishn Formation [63]. Detailed facies descriptions and palaeo-environmental interpretations of the Qishn Formation in this area can be found in Sena et al. [63]. In the offshore Arabian Gulf, packstone to wackestone and finely crystalline dolomite layers were collected from the free-water level reservoir zone in the Barremian–Aptian, Kharaib Formation. The detailed facies description in this interval can be found in [64] and the palaeo-environmental interpretation of this interval can be found in Van Buchem et al. [44] and Yamamoto et al. [65]. The dolomite in this formation is most abundant in the lower two-thirds of the interval [66].

The Jurassic carbonates were obtained from two locations: the Musandam Peninsula and the Jabal Akhdar (Figure 2). In the Musandam Peninsula, microbial laminate mudstone was collected from the Musandam II Formation outcrop in Wadi Naqab [60]. The dolomite was collected from the dolomitised burrow infilling of the mudstone of the Musandam II Formation outcrop. Facies description and palaeo-environmental interpretation of this formation were previously published [60]. In the Jabal Akhdar, Central Oman Mountains, grey limestone and brown strata-bound dolomite samples were collected from the Jurassic Sahtan Group outcrop [59]. The limestone bed has a mudstone to wackestone texture [59]. The stratabound replacive dolomites in this area are interpreted to have formed relatively early [59].

Permian outcrop samples were collected ~1 km away from the Jurassic samples, towards the centre of the Jabal Akhdar dome. Finely laminated limestones and finely crystalline brown replacive dolomites were collected from the Saiq Formation outcrop [36].

Calcites and dolomites were sampled from the fine grain matrix. In an attempt to collect single mineral phases, cut surfaces were sampled using a dental drill at 1000–2000 rpm. To obtain a homogeneous powder size, the samples were then gently crushed by hand in an agate mortar and pestle and then passed through a 125 µm sieve.

3.2. Petrography

Petrographic observations were made using a transmitted light Zeiss Axioskop 40 (Zeiss, Leipzig, Germany), followed by cathodoluminescence (CL) microscopy using a Nikon Eclipse 50i microscope (Nikon, Tokyo, Japan) equipped with a CITL cathodoluminescence MK5-2 stage. The operating conditions for the CL stage were a current of 315 µA, an accelerating voltage of 15 kV, and a vacuum operating at 0.003 mBar.

3.3. FTIR Mineralogy and X-ray Diffraction Analysis of Dolomite

Fourier transform infrared spectroscopy (FT-IR-Spectrometer Nicolet 5700, Thermo Fisher, Bremen, Germany) was applied to all samples to screen for mineralogical homogeneity and to qualitatively estimate mineral content (calcite or dolomite) following reference [67].

X-ray diffraction characterisation of dolomite samples focused on quantification of dolomite abundance relative to calcite, dolomite stoichiometry, and dolomite cation ordering [68]. Measurements of stoichiometry and cation ordering were carried out in the Carbonate Petrology and Characterisation Laboratory at Western Michigan University. Measurements were performed using a Bruker D2 Phaser Diffractometer equipped with a CuK α anode, using a 2θ range of 20–60°, a step size of 0.018°/step, and a count time of 1 s/step. Peak positions were calibrated using powdered fluorite (CaF₂) as an internal standard [69].

The percentage of dolomite relative to calcite was calculated using the background-corrected intensity ratio of the dolomite {104} reflection to the sum of the intensities of the dolomite {104} and calcite {104} reflections [70]. Dolomite stoichiometry was calculated using the calibrated position of the dolomite {104} reflection, consistent with the approach developed by Lumsden [71]. All values of dolomite stoichiometry in the database are reported as mole% MgCO₃. The degree of cation ordering was determined using the ratio of the background-subtracted intensities of the dolomite {015} and {110} reflections following the methods of Goldsmith and Graf [72,73].

3.4. Clumped Isotopes

Clumped isotopes and $\delta^{18}\text{O}$ 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, following the method described by Adlan and John [33] and Adlan et al. [74]. The standard-to-samples ratio used in our study is one standard for every three sample measurements. Mass 48 and mass 49 signal spikes were monitored for each measurement to detect hydrocarbons, chlorocarbons [75,76], and sulphur-bearing contaminants [76,77]. To be deemed clean, a replicate analysis needed a Δ_{48} offset value < 1.5‰ and/or a 49 parameter value < 0.3 [78].

Calculations and corrections of raw Δ_{47} , $\delta^{18}\text{O}$, and $\delta^{13}\text{C}$ were performed using the free software Easotope [79]. Non-linearity in the mass spectrometer was corrected with the pressure baseline correction methods developed at the ETH Zurich [80], and the Δ_{47} values projected in the inter-laboratory absolute reference frame or Intercarb Carbon Dioxide Equilibrated Scale (I-CDES) [81] based on our routinely measured ETH1, ETH2, ETH3, ETH4, and our internal Carrara marble (ICM) carbonate standards [81,82]. This Δ_{47} (I-CDES) was used to calculate the temperature of (re)crystallisation for the carbonates by applying a recent multi-lab calibration [83].

The conventional $\delta^{18}\text{O}$ value was corrected for acid digestion at 90 °C by multiplying the $^{18}\text{O}/^{16}\text{O}$ ratio by the alpha value of 1.0081 based on the published fractionation factor of [84] for calcite and 1.0093 for dolomite [85].

3.5. Modelling

A forward solid-state reordering model was created to test various geological scenarios in order to explain our calcite and dolomite record of Δ_{47} values and also to demonstrate the benefits of reconstructing thermal history using co-occurring calcite and early dolomite. Two models were examined based on the competing burial/exhumation scenarios. For the Arabian Gulf burial scenario, the model assumes an initial mineralisation temperature for calcite and dolomite of 25 °C (Δ_{47} = 0.590‰ with 0.01‰ uncertainty), compatible with estimates for equatorial sea surface paleotemperatures [86]. Based on the present-day formation temperature, a final temperature of 130 °C (Δ_{47} = 0.394‰) was assumed reflecting the maximum burial depth. Reordering models were simulated for a 125 Ma burial period initialised from 0 to 125 timescales, respectively, according to the depositional

ages of the interval (Barremian). All calculations were performed using *isotopylog*, an online accessible open-source package in Python 3.7 [87]. In contrast, the Jabal Akhdar exhumation scenario assumes an initial peak burial temperature for calcite and dolomite of 280 °C ($\Delta_{47} = 0.277\text{‰}$). Based on present-day surface temperatures, an end temperature of 25 °C ($\Delta_{47} = 0.590\text{‰}$) was used. A previous study suggested that the uplift process, including its dome formation with the steady and rapid exhumation on Jabal Akhdar, was initiated at ~80 Ma to the present day [39]. Thus, the reordering models were simulated for the past 80 million years.

The models tested the paired-diffusion model equation [7] on calcite minerals and the disordered kinetic model equation [17] for both calcite and dolomite. The disordered kinetic equation was selected as it is a more adaptable kinetic model built based on multiple laboratory results [17]. Also, it was previously theoretically shown that all previous kinetic models are specific cases of disordered kinetics [17]. The paired-diffusion model equation is used here as a comparison as it is the most prominent equation known for thermal history modelling. The calcite mineral in the paired-diffusion model [7] uses a specific activation energy (μ_E) of $250.7 \pm 13.6 \text{ kJ mol}^{-1}$ and a pre-exponential factor (ν_0) of $34.2 \pm 2.3 \text{ min}^{-1}$ [17], while the disordered kinetic model uses an activation energy (μ_E) of $224.3 \pm 27.6 \text{ kJ mol}^{-1}$ and a pre-exponential factor (ν_0) of $31.5 \pm 4.6 \text{ min}^{-1}$ [17]. The dolomite in the disordered kinetic model uses a specific activation energy (μ_E) of $230.3 \pm 47.7 \text{ kJ mol}^{-1}$ and a pre-exponential factor (ν_0) of $29.0 \pm 6.8 \text{ min}^{-1}$ [17]. This paired reaction–diffusion model attributes the change in Δ_{47} to the neighboring singly substituted carbonate group pair interactions, where the group can then diffuse through the crystal lattice to exchange isotopes [7]. On the other hand, the recent adaptable disordered kinetic model attributed the change in Δ_{47} as random-walk ^{18}O diffusion, which increases the reordering rates naturally through the carbonate lattice [17]. The model predicts Δ_{47} values at sequential time steps of the imposed temperature–time (T–t) histories. The predicted Δ_{47} values were then compared to the measured Δ_{47} in the samples. Details of the kinetic model derivation, including the Arrhenius parameters adaptation, are described in [17]. The final predicted Δ_{47} values for the present day were then directly compared to the measured Δ_{47} of our samples. The temperatures from Δ_{47} values were then calculated using the equation of [83].

4. Results

4.1. Petrography Observations

A cathodoluminescence photomicrograph of the limestone shows that the Cretaceous samples in Area 2 (see Table 1) consist of wackestone to packstone textures with limpid sparite crystal replacement inside the skeletal fragment with a micritic matrix (Figure 3A). Figure 3A shows dark luminescence-sparitic patches or equant-blocky calcite within the interparticle porosity and also in the skeletal remnant as a replacement. The micrite in the Cretaceous limestone exhibits a range of dark to bright red CL (Figure 3A). Towards the deeper burial realm of Area 3, the Jurassic interval consists of micritised-oid wackestone with medium-sized dolomite rhombohedra (10–30 μm in diameter) hosted in a calcite cement matrix (Figure 3B). Figure 3B shows that the replacement of dolomite patches is recognised inside ooid nuclei. A dedolomite (calcite replacing dolomite) is recognised in some of the dolomite cement (Figure 4B). In the deepest burial realm of Area 4, the Jurassic interval consists of micrite-dominated mudstone–wackestone with patches of sparite or blocky calcite crystals within clasts and skeletal remnants (Figure 3C). In contrast, the Permian interval consists of micritic mudstone and wackestone with equant-blocky calcite crystals within skeletal remnants (Figure 3D). The equant-blocky calcite observed in the Jurassic and Permian samples in Area 4 exhibits non-luminescence (Figure 3C,D).

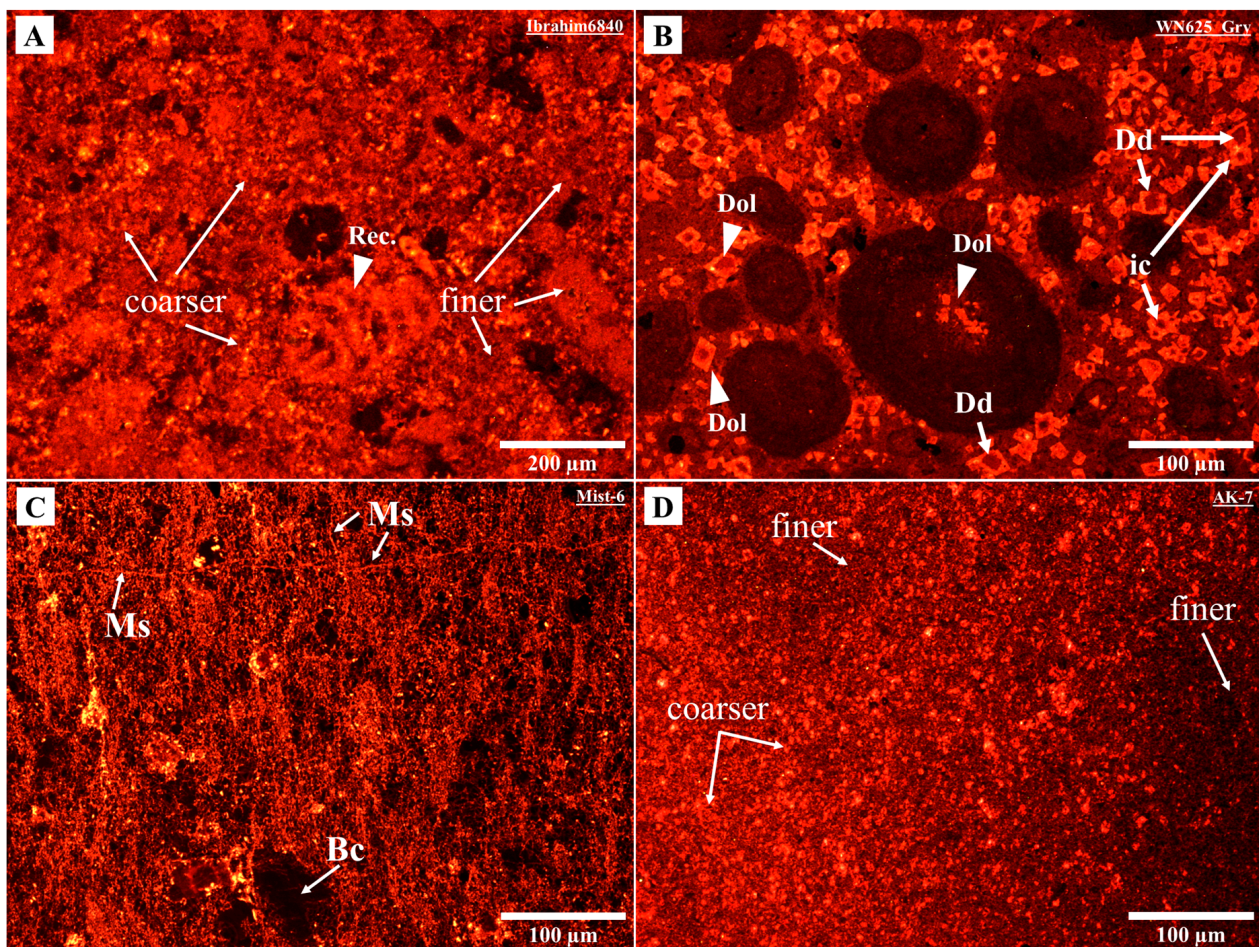


Figure 3. Limestone photomicrographs with cold cathodoluminescence. (A) Ibrahim6840 Cretaceous sample from Area 2 comprises wackestone–packstone texture, recrystallised skeletal grain, and inhomogeneous crystal-sized micrite with series of dark to bright red luminescence. (B) WN625_Gry Jurassic sample from Area 3 comprises micritised-oids wackestone with dolomite rhombs floating in a calcite cement matrix and ooids. A trace of calcitification (dull luminescence) is recognised inside dolomite rhombs. (C) Mist-6 Jurassic sample from Area 4 comprises micrite-dominated mudstone–wackestone with patch of sparite or equant-blocky calcite crystal. (D) Ak-7 Permian sample from Area 4 consists of mudstone with inhomogeneous crystal-sized micrite. ‘Rec.’: recrystallised form; ‘Dol’: dolomite cement; ‘Ms’: micro-seam fracture; ‘ic’: interlocking contact; ‘Bc’: equant-blocky calcite.

Cretaceous dolomites in Area 2 are characterised by fine to medium nonplanar-a to planar-e dolomite crystals that have irregular or extensively interlocking contacts between rhombs (Figure 4A). In Area 3, variation in dolomite crystals in the Jurassic interval is recognised from fine to medium nonplanar-a with interlocking contacts to planar-e dolomite with a cloudy/sucrosic texture of fine, brown crystals (Figure 4B). The dolomite nuclei observed in Area 3 are characterised by dedolomite with a dull CL signature. In the deepest burial stage of Area 4, the Jurassic interval consists of medium planar-e dolomite crystals with intensive fine-micritic dolomite (Figure 4C). Irregularity on the outer surface of the crystals, similar to a dissolution pattern, is present on the dolomite rhombs (Figure 4C). The Permian interval in Area 4 consists of fine to medium planar-e to nonplanar-a dolomite crystals with a bright outer rim and a darker interior (Figure 4D).

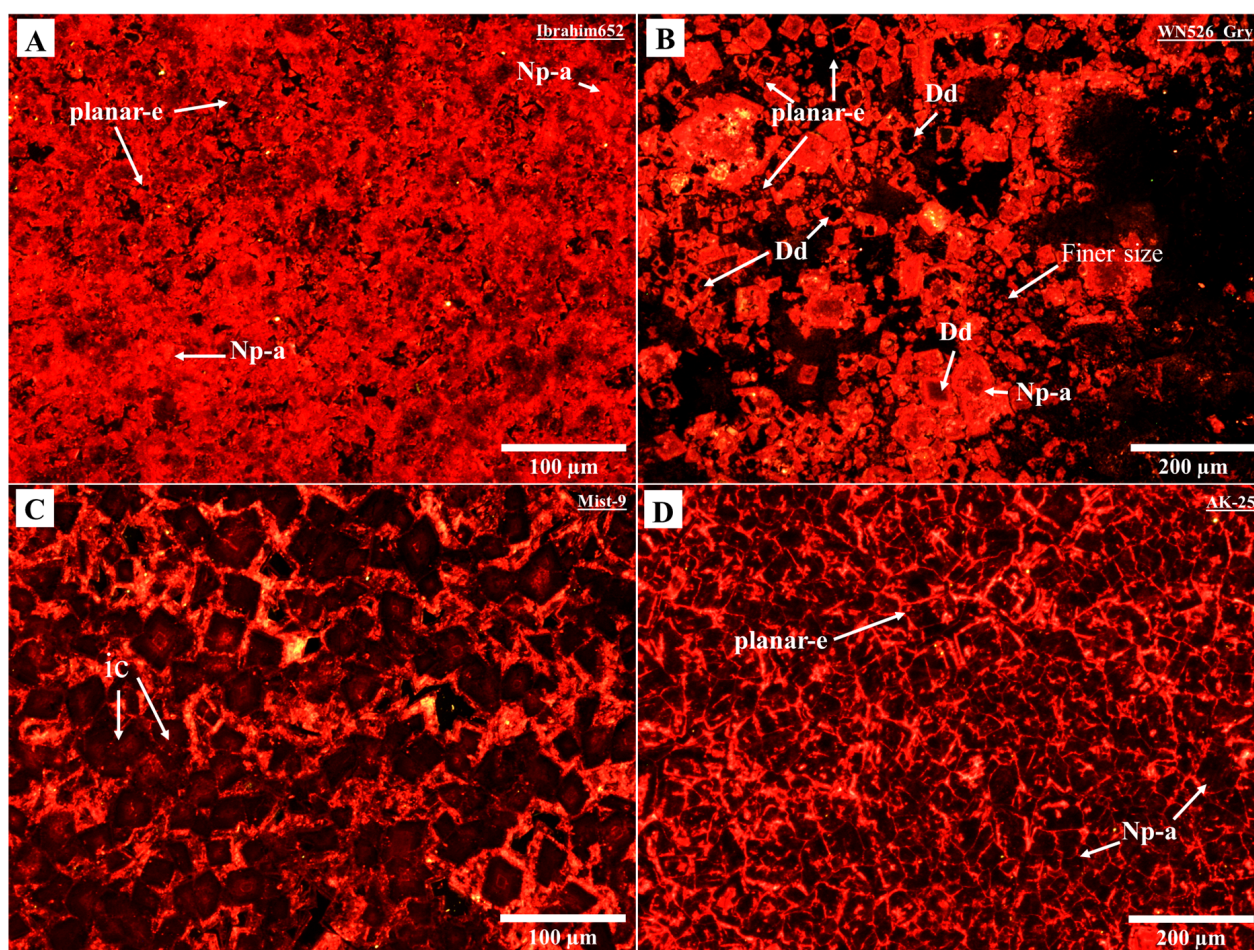


Figure 4. Dolomite photomicrographs with cold cathodoluminescence. (A) Ibrahim652 Cretaceous sample from Area 2 consists of fine to medium nonplanar-a to planar-e dolomite crystals with irregular or extensively interlocking contact. (B) WN526_Gry Jurassic sample from Area 3 consists of fine to medium nonplanar-a to planar-e dolomite crystals with trace of calcitification/dedolomitisation (dull luminescence) recognised inside rhomb nuclei. (C) Mist-9 Jurassic sample from Area 4 comprises dark luminescent planar-e to planar-s dolomite with interlocking crystals. The micritic matrix appears to have brighter luminescence compared to the medium-sized rhomb crystals. (D) Ak-25 Permian samples from Area 4 consist of fine to medium planar-e to nonplanar-a dolomite crystals interlocking each other. ‘Dd’: dedolomitisation; ‘ic’: interlocking contact; ‘Np-a’: nonplanar-a.

4.2. $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ Results

The conventional bulk isotope compositions of calcite and early dolomite (Table 2) are summarised in Figure 5A. From shallow to deep burial depths, $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ increase from Area 1 towards Area 4 (Figure 5A). The $\delta^{18}\text{O}$ values measured in Cretaceous calcite in Area 1 is -4.87‰ , with dolomite ranging from -5.78 to -0.31‰ , whereas the $\delta^{13}\text{C}$ value is 1.34‰ for calcite, with a range of -0.04 to 1.70‰ for dolomite (Table 2). In Area 2, the $\delta^{18}\text{O}$ values of Cretaceous calcite range from -8.37 to -4.86‰ and dolomite values range from -6.33 to 6.5‰ . In the same area, $\delta^{13}\text{C}$ values range from 2.92 to 3.43‰ , and dolomite values range from 4.61 to 4.85‰ (Table 2).

Table 2. Carbonate clumped isotope values (Δ_{47}) and conventional isotope composition ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) of areas used in the study, including its mineralogical information.

Sample Information	Mineralogy-FTIR ^(a)	Depth ^(b) (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. ^(c) (‰, SMOW)	$\delta^{18}\text{O}$ fl. SE (‰)	Δ_{47} (‰, I-CDES)	Δ_{47} SE (‰)	Δ_{47} Temp. ^(d) (°C)	Δ_{47} Temp. SE (°C)
Area 1 Haushi-Huqf outcrop, Oman													
Cretaceous													
SGF37 ^(e)	Calcite	400	3	1.34	0.04	−4.87	0.02	2.08	0.36	0.535	0.008	47	2
E37	Dolomite	400	3	1.70	0.01	−5.78	0.04	2.00	0.69	0.522	0.009	53	2
I5	Dolomite	400	3	−0.04	0.03	−0.31	0.13	6.22	0.97	0.541	0.012	45	2
Area 2 offshore, subsurface core, Arabian Gulf													
Cretaceous													
Z94708-m ^(f)	Calcite	2886	3	3.14	0.09	−7.04	0.18	4.17	0.86	0.478	0.012	74	7
Z94708-sk ^(f)	Calcite	2886	3	3.09	0.10	−6.83	0.05	3.11	0.56	0.494	0.007	66	4
Ibrahim484M	Calcite	2950	4	3.40	0.20	−6.36	0.05	4.09	0.57	0.488	0.008	69	4
Ibrahim484R	Calcite	2950	3	3.43	0.03	−7.11	0.07	6.24	0.52	0.452	0.005	89	3
Ibrahim6840	Calcite	2950	4	2.92	0.04	−4.86	0.06	4.39	0.40	0.504	0.006	61	3
Ibrahim708S	Calcite	2950	3	3.23	0.03	−8.37	0.08	9.43	0.70	0.400	0.007	125	6
Ibrahim652	Dolomite	2943	5	4.61	0.16	−6.5	0.09	12.63	1.13	0.387	0.013	136	11
Ibrahim653	Dolomite	2943	3	4.85	0.01	−6.33	0.05	14.62	0.60	0.368	0.005	154	5
Area 3 Wadi Naqab outcrop, Musandam Peninsula													
Jurassic													
WN526	Calcite	5100	3	−3.97	0.02	−2.90	0.06	8.37	0.44	0.479	0.005	74	3
WN453_Gry	Calcite	5100	4	−0.27	0.02	−3.67	0.02	10.71	1.61	0.441	0.019	96	13
WN627	Calcite	5100	3	2.01	0.05	−3.03	0.06	16.68	1.27	0.382	0.013	141	12
WN625_Gry	Calcite	5100	4	2.32	0.16	−2.74	0.13	20.36	1.25	0.348	0.013	176	15
WN625_Red	Dolomite	5100	5	2.30	0.13	−3.99	0.22	16.55	1.20	0.373	0.012	150	11
WN526_Gry	Dolomite	5100	4	0.09	0.04	−3.17	0.11	19.81	0.94	0.349	0.008	175	10
WN453_Wsk	Calcite	5100	3	1.75	0.03	−4.24	0.07	19.33	1.26	0.343	0.012	182	15
Area 4 Jabal Akhdar outcrop, Central Oman Mountain													
Jurassic													
Mist-2	Calcite	8000	3	0.05	0.00	−8.14	0.03	9.82	1.10	0.399	0.012	126	10
Mist-6	Calcite	8000	3	1.27	0.01	−4.50	0.02	15.74	1.30	0.376	0.014	147	13
Mist-5	Dolomite	8000	3	0.52	0.03	−5.93	0.09	10.37	0.43	0.418	0.004	112	3
Mist-9	Dolomite	8000	3	1.73	0.04	−3.38	0.01	15.27	1.31	0.393	0.014	131	12
Mist-31	Dolomite	8000	3	2.03	0.01	−5.34	0.05	16.05	0.45	0.364	0.004	158	4
Mist-8	Dolomite	8000	3	1.69	0.02	−7.35	0.11	18.28	1.28	0.323	0.011	208	16
Permian													
MPA11A	Calcite	9000	5	2.41	0.02	−6.13	0.07	13.48	1.04	0.382	0.011	141	10
MPA11B	Calcite	9000	4	3.36	0.31	−7.49	0.51	6.69	3.27	0.442	0.033	95	22
WSNE2_C	Calcite	9000	4	2.00	0.06	−6.40	0.16	13.02	1.41	0.384	0.014	139	12
AK-13	Calcite	9000	3	6.04	0.09	−3.35	0.10	19.94	0.05	0.346	0.001	178	1
AK-7	Calcite	9000	3	5.46	0.01	−2.82	0.02	19.65	0.76	0.354	0.013	169	9
MPA35	Dolomite	9000	3	6.38	0.03	−0.58	0.05	18.73	0.97	0.387	0.010	136	9

Table 2. Cont.

Sample Information	Mineralogy-FTIR ^(a)	Depth ^(b) (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. ^(c) (‰, SMOW)	$\delta^{18}\text{O}$ fl. SE (‰)	Δ_{47} (‰, I-CDES)	Δ_{47} SE (‰)	Δ_{47} Temp. ^(d) (°C)	Δ_{47} Temp. SE (°C)
AK-23	Dolomite	9000	3	6.08	0.01	−1.06	0.01	24.99	0.67	0.321	0.006	211	9
AK-24	Dolomite	9000	3	5.92	0.05	−1.05	0.05	22.10	0.04	0.348	0	176	1
AK-25	Dolomite	9000	3	6.02	0.01	−0.02	0.04	26.96	1.65	0.313	0.014	223	22
AK-27	Dolomite	9000	3	3.21	0.03	0.38	0.09	22.45	1.29	0.359	0.012	164	13

^(a) Fourier-transform infrared spectroscopy mineralogy after Henry et al. [67]. ^(b) Estimated maximum burial stratigraphic depth of Jabal Akhdar Central Oman Mountains obtained from Grobe et al. [39]. Distance between Jurassic and Permian sampling location is ~1km; The maximum burial depth of Musandam Peninsula obtained from Carminati et al. [38]; Haushi-Huqf burial from Immenhauser et al. [40] and Sattler et al. [51]. ^(c) Calculated diagenetic fluid $\delta^{18}\text{O}$ SMOW from calcite $\delta^{18}\text{O}$ using Kim and O'Neil [88] equation. ^(d) The calculated temperature from Δ_{47} value using Anderson et al. [83] equation. ^(e) Cretaceous calcite sample with maximum temperature record measured by Patel [89]. ^(f) Calcite samples from Adlan and John [33].

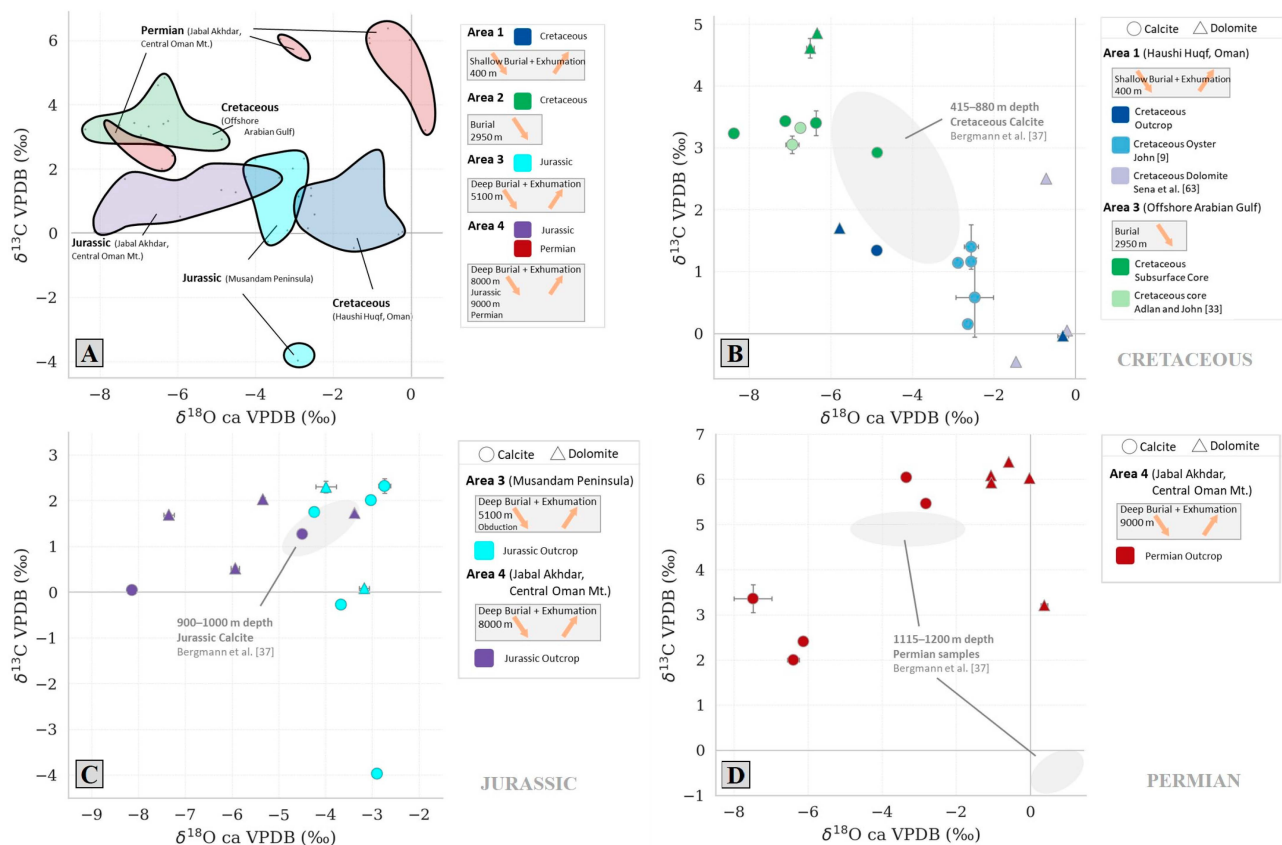


Figure 5. Cross-plot of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ of each area used in this study. (A) Comparison of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ composition, including the burial history information on each area. Calcite and early dolomite are labelled in order to best highlight trends in geographic areas and sample ages. (B) Cretaceous samples from an outcrop in Haushi-Huqf and subsurface core in the offshore Arabian Gulf. Published bulk isotope composition from Sena et al. [63], John [9], and Adlan and John [33] are also shown. (C) Jurassic samples from an outcrop in the Musandam Peninsula and Jabal Akhdar area, Central Oman Mountains. (D) Permian samples from Central Oman Mountains. Within figures (B–D), the Cretaceous, Jurassic, and Permian subsurface samples from clumped isotopes studied in Central Oman High (HM well; HM-13-1 at 1115 m depth and HM-14-1 at 1199 m) by Bergmann et al. [37] are also shown.

The $\delta^{18}\text{O}$ values of Jurassic calcite in Area 3 range from -4.24 to -2.74 ‰ (Figure 5C), with dolomite ranging from -3.99 to -3.17 ‰. The calcite $\delta^{13}\text{C}$ values range from -3.97 to 2.32 ‰, with dolomite ranging from 0.09 to 2.30 ‰ (Table 2). The $\delta^{18}\text{O}$ values of Jurassic calcite in Area 4 range from -8.14 to -4.50 ‰ (Figure 5C), with dolomite ranging from -7.35 to -3.38 ‰, whereas the calcite's $\delta^{13}\text{C}$ values range from 0.05 ‰ to 1.27 ‰, with dolomite ranging from 0.52 to 2.03 ‰ (Table 2 and Figure 5C). In the same Area 4, the $\delta^{18}\text{O}$ values of Permian calcite range from -7.49 to 2.82 ‰, with dolomite ranging from -1.06 to 0.38 ‰, whereas the calcite's $\delta^{13}\text{C}$ values range from 2.00 to 6.04 ‰, with dolomite ranging from 3.21 to 6.38 ‰ (Table 2 and Figure 5D).

4.3. Clumped Isotope (Δ_{47})

The Δ_{47} values measured in Cretaceous calcite are 0.535 ‰ in Area 1 (Haushi-Huqf), whereas the dolomite values range from 0.522 to 0.541 ‰. The overall Δ_{47} values of the Cretaceous carbonates decrease toward the offshore Arabian Gulf region, Area 2, with values ranging from 0.400 to 0.504 ‰ for calcite and from 0.368 to 0.387 ‰ for dolomite (Table 2). The calcites from the Arabian Gulf have higher Δ_{47} values compared to dolomites co-located in the same stratigraphic interval.

The Δ_{47} values of Jurassic calcite in Area 3 (Musandam Peninsula) range from 0.343 to 0.479‰, whereas the dolomite values range from 0.349 to 0.373‰ (Table 2). In Area 4 (Jabal Akhdar), the Δ_{47} values of Jurassic calcite range from 0.376 to 0.399‰, whereas the dolomite values range from 0.323 to 0.418‰ (Table 2). The Δ_{47} values of Permian calcite in Jabal Akhdar range from 0.346 to 0.442‰, whereas the dolomite values range from 0.313 to 0.387‰ (Table 2). In this area, both Jurassic and Permian calcites have higher Δ_{47} values compared to co-located dolomites in the same stratigraphic interval.

The maximum burial depth reached by the carbonates increases from Area 1 towards Area 4 and is reflected in the higher clumped isotope temperatures ($T_{\Delta_{47}}$) reported in Table 2. The calculated clumped isotope temperature for the Cretaceous calcite in Area 1 of Haushi-Huqf is 47 °C, whereas the dolomite values range from 45 °C to 53 °C (Table 2). The calcite temperature in Area 2 of the offshore Arabian Gulf ranges from 61 °C to 125 °C, whereas the dolomite values range from 136 °C to 154 °C (Table 2). In Area 3 (Musandam Peninsula), Jurassic calcite temperatures range from 74 °C to 182 °C, whereas the dolomite values range from 150 °C to 175 °C (Table 2). In Area 4 (Jabal Akhdar), Jurassic calcite temperatures range from 126 °C to 147 °C, whereas the dolomite values range from 112 °C to 208 °C (Table 2). In the same area, Permian calcite temperatures range from 95 °C to 178 °C, whereas the dolomite values range from 136 °C to 223 °C (Table 2).

5. Discussion

5.1. Evidence of Recrystallisation

In all areas investigated, typical burial signatures were recognised in thin sections, including mechanical compaction as evidenced by interlocking crystal contacts (Figures 3B and 4C), the mineral replacement of skeletal fragments (Figure 3A), non-luminescent calcite and inclusion-rich dolomite cements (Figures 3A,C and 4A,C,D), and microfractures (Figure 3C).

A unique recrystallisation pattern occurred in Area 2, including different crystal sizes and skeletal grain brightness, and the inhomogeneous crystal size of the micrite (Figure 3A). This micrite size variation (enlargement) is a consistent very deep burial diagenesis signature [90] and was previously reported in the region [91]. Recent experiments by Hashim and Kaczmarek [92] showed that calcite precipitation is unlikely to occur in marine diagenetic environments. Collectively, these observations suggest that the variation in calcite crystal morphology is most consistent with the alteration signature caused by burial compaction and cementation [93–95].

For the dolomite in Area 2, the distribution of the micritic-fine crystals (Figure 4A) appears to disregard pre-existing crystal boundaries, implying recrystallisation [96]. Most of the dolomites selected for this study are inclusion-rich (Figure 4), which is also consistent with recrystallisation [96–98]. The presence of a range of luminescence in fine-grained textures of both dolomite and calcite is interpreted here as an indication of recrystallisation [90,99].

Burial diagenesis was also recognised in the $\delta^{18}\text{O}$ values in some areas. In the Cretaceous samples, for example, progressively depleted $\delta^{18}\text{O}$ values are observed with increasing depth (Figure 5B, Area 1 towards Area 2), an observation consistent with diagenesis during progressive burial [100–102]. A similar trend in the $\delta^{18}\text{O}$ values in deeper burial depths was also observed in the Jurassic calcite and dolomite (Figure 5C, Area 3 towards Area 4). This pattern is consistent with temperature-dependent $\delta^{18}\text{O}$ depletion on carbonate minerals at elevated temperatures [103–105].

5.2. Water–Rock Ratio during Recrystallisation Process

In general, the $\delta^{18}\text{O}$ values of the recrystallised calcites and dolomites of each area (Figure 5A) exhibit a trend of progressively more negative values in response to temperature elevation, consistent with previous studies [106–108]. The $\delta^{18}\text{O}$ values of recrystallised minerals are controlled by the temperature-dependent fractionation of oxygen isotopes between mineral and fluid [106,109–112]. Thus, it is crucial to analyse the oxygen isotopic composition of the diagenetic fluid ($\delta^{18}\text{O}_{\text{fluid}}$) in order to understand the environment in

which the mineral precipitated. This is possible by combining the Δ_{47} temperature with the oxygen isotopic composition of the carbonates ($\delta^{18}\text{O}_{\text{Ca}}$) to calculate $\delta^{18}\text{O}_{\text{fluid}}$ (Table 2) using the equations for calcite [88] and dolomite [113]. Figure 6A shows that the $\delta^{18}\text{O}_{\text{fluid}}$ evolves towards a more positive composition with increasing temperature, corresponding to a deeper burial regime (Haushi-Huqf area towards Jabal Akhdar area).

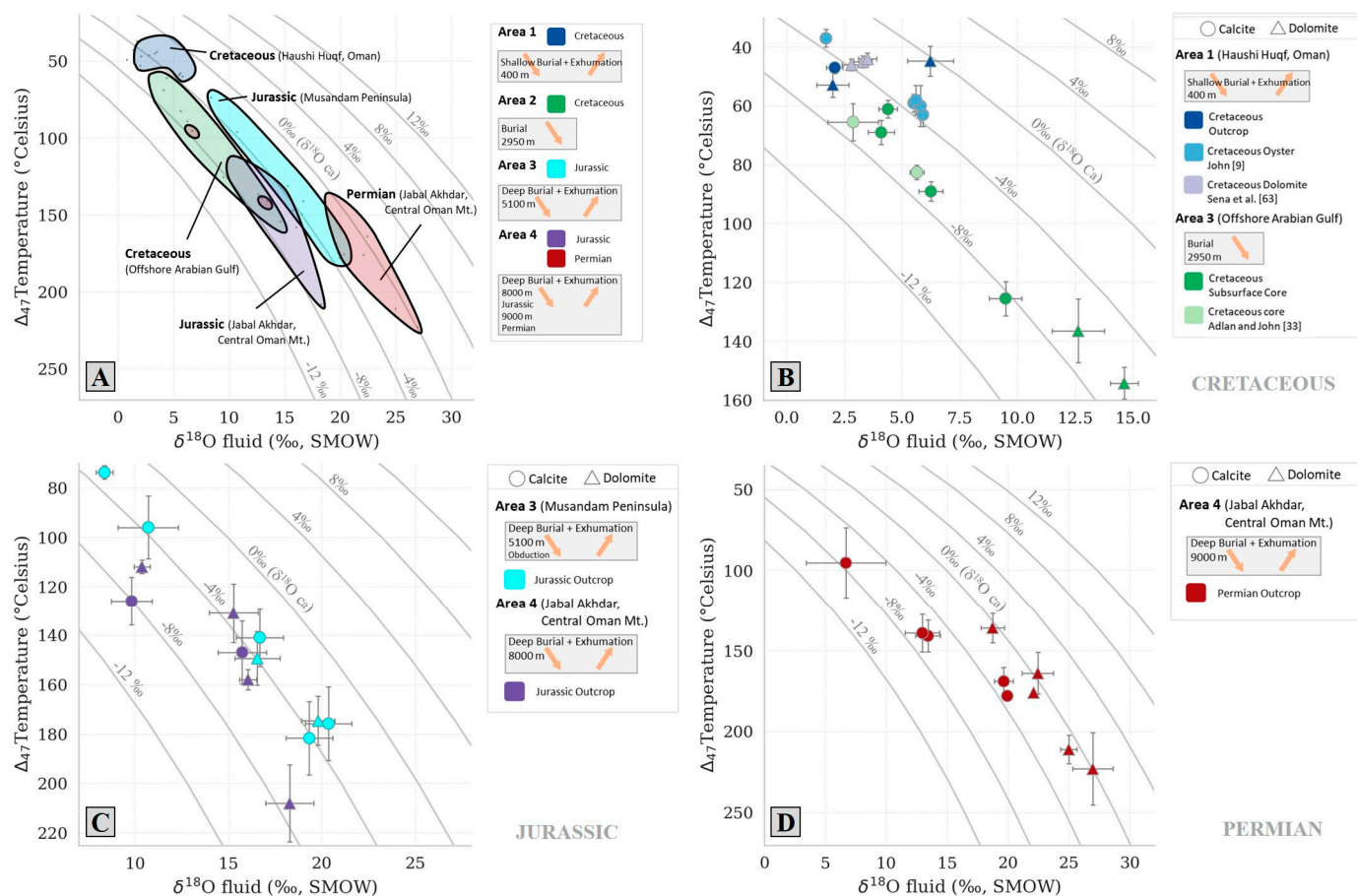


Figure 6. The Δ_{47} temperature versus the calculated $\delta^{18}\text{O}$ of the diagenetic fluid. Values were reconstructed using the Friedman and O’Neil [104] equation for calcite and the Horita [113] equation for dolomite. The isolines showed the mineral composition ($\delta^{18}\text{O}_{\text{Ca}}$) of the samples. (A) Overall data according to area, including the burial history information. (B) Cretaceous samples from an outcrop in Haushi-Huqf and subsurface core in the offshore Arabian Gulf. Published bulk isotope composition from Sena et al. [63], John [9], and Adlan and John [33] are also shown. (C) Jurassic samples from outcrops in the Musandam Peninsula and Jabal Akhdar. (D) Permian samples from outcrop in Jabal Akhdar.

Area 1 has the shallowest burial depth in our study. The calcite recrystallisation patterns here are similar to the previous reports on recrystallised oyster shells with nearly invariant $\delta^{18}\text{O}_{\text{Ca}}$ [9], suggesting a low water–rock ratio. However, the wide variability in the $\delta^{18}\text{O}_{\text{Ca}}$ of the dolomite under a relatively constant temperature suggests a near-surface diagenetic influence on the samples.

Towards the deeper areas, the remainder of our samples appear to have recrystallised with an invariant $\delta^{18}\text{O}_{\text{Ca}}$ mineral composition with a wide temperature evolution (Δ_{47}). In Area 2, the $\delta^{18}\text{O}$ values of Cretaceous calcite and dolomite remain remarkably invariant at $-6.67 \pm 0.9\text{‰}$, with a temperature range from 61 °C to 125 °C for calcite and 136 °C to 154 °C for dolomite (Figure 6B). In Area 3 and Area 4, the $\delta^{18}\text{O}$ values of Jurassic calcite and dolomite were also relatively invariant at $-3.39 \pm 0.5\text{‰}$ and slightly lighter in Area 5 at $-5.57 \pm 1.6\text{‰}$ (Figure 6C). Interestingly, the $\delta^{18}\text{O}$ values of the Permian samples in Area 4

are similar to Permian samples in Central Oman High [37], with invariant calcite $\delta^{18}\text{O}$ values at $-5.24 \pm 1.8\text{‰}$ and the heavier $\delta^{18}\text{O}$ for dolomite at $-0.47 \pm 0.5\text{‰}$ (Figure 6D).

Together with the constant $\delta^{18}\text{O}$ values, trends of diagenetic fluid evolution are clearly recognised (Figure 6B,C), from low temperatures and lighter $\delta^{18}\text{O}_{\text{fluid}}$ to high temperatures and heavy $\delta^{18}\text{O}_{\text{fluid}}$. We interpret these results to reflect the diagenetic alteration (i.e., the recrystallisation) of the calcites and dolomites in all areas occurring in low water–rock ratio conditions or in a rock-buffered systems that might involve solid-state reordering, where the isotopic composition of carbonate remains relatively invariant despite burial and exhumation.

5.3. Calcite vs. Dolomite Temperature

Both the average and maximum Δ_{47} temperatures of dolomites are higher than calcite across a range of maximum burial depths (Table 3 and Figure 7), with the exception of data from the Musandam Peninsula. In this area, the similarity between the maximum Δ_{47} temperature recorded for dolomite and calcite are attributed to dedolomitisation, which can account for the low dolomite abundance (<35%) found in the sample (Figure 4B). In Haushi-Huqf, the difference between the maximum Δ_{47} temperatures recorded in Cretaceous dolomite and calcite is not significant or not prominent (5 °C, with 1 Standard Error of 4 °C), which can be explained by the shallow burial history [40,51], which is inadequate to trigger Δ_{47} reordering as the maximum temperature is not high enough. The temperature disparity becomes more expansive with the increasing maximum burial estimates of the samples, resulting in a trend toward an increasing temperature regime (Figure 7). In this figure, the difference between the maximum Δ_{47} temperature recorded in Cretaceous dolomite and calcite offshore in the Arabian Gulf is 29 ± 17 °C, which is significantly more expanded in the Jurassic rock in Jabal Akhdar (61 ± 23 °C) and the Permian rock of the Jabal Akhdar (45 ± 19 °C). These results provide empirical evidence from the rock record that calcite and dolomite behave differently during recrystallisation, which could result in different Δ_{47} records despite them having the same thermal history. This is consistent with lab experiments showing that different carbonate minerals have different C–O bond reordering kinetics, i.e., they have different Arrhenius parameters and variations in frequency factors [16,17,21]. As the minerals experience substantial burial heating, the kinetic difference will result in a greater impact on recorded Δ_{47} temperature depending on the cooling rates.

Table 3. The Δ_{47} temperature information of dolomite and calcite samples in each area, the maximum burial reached, and the temperature disparity between dolomite and calcite.

Sample Information	Depth ^(a) (m)	G.Geo. Temp. ^(b) (°C)	Δ_{47} Temp. ^(c) (°C)						Δ_{47} Temp. ^(d) (°C) Dol. – Cal.	S.E. Temp. Dol. – Cal.
			n	Mean	S.E.	Median	Min	Max		
<i>This Study</i>										
Haushi-Huqf outcrop, Oman (Area 1)										
Cretaceous Calcite ^(e)	400	47.5	24	34	4	35	21	47	5	4
Dolomite ^(f)	400	47.5	5	47	2	45	44	53		
Offshore, subsurface core, Arabian Gulf (Area 2)										
Cretaceous Calcite	2950	130.0	4	86	14	79	61	126	29	17
Dolomite	2943	130.0	2	145	9	145	136	154		
Wadi Naqab outcrop, Musandam Peninsula (Area 3)										
Jurassic Calcite	5100	168.4	4	122	23	118	74	176	−1	26
Dolomite	5100	168.4	2	162	13	162	149	175		

Table 3. Cont.

Sample Information	Depth ^(a) (m)	G.Geo. Temp. ^(b) (°C)	Δ_{47} Temp. ^(c) (°C)						Δ_{47} Temp. ^(d) (°C) Dol. – Cal.	S.E. Temp. Dol. – Cal.
			n	Mean	S.E.	Median	Min	Max		
Jabal Akhdar outcrop, Central Oman Mountain (Area 4)										
Jurassic									61	23
Calcite	8000	169.3	2	137	11	137	126	147		
Dolomite	8000	169.3	4	152	21	145	112	208		
Permian									45	19
Calcite	9000	187	5	145	14	141	95	178		
Dolomite	9000	187	5	181	13	177	136	223		
Other studies										
Central Oman High, subsurface core, Oman (Bergmann et al. [37])										
Permian									6	11
Calcite	1115	55.3	18	36	3	35	30	43		
Dolomite	1199	56.3	3	36	11	33	28	49		
Leached samples from Mx13 well in Sichuan Basin, China (Lu et al. [114])										
Permian/Triassic									–36	14
Calcite	3556–3899	~113	2	119	11	119	117	120		
Dolomite	3556–3899	~113	2	79	11	79	74	83		
Cambrian									–46	30
Calcite	4370–4601	~140	3	130	21	134	121	136		
Dolomite	4370–4601	~140	3	81	21	83	71	90		

^(a) The maximum burial stratigraphic depth of Jabal Akhdar Central Oman Mountains obtained from Grobe et al. [39], distance between Jurassic and Permian sampling location is ~1000 m; The maximum burial depth of Musandam Peninsula obtained from Carminati et al. [38]; Haushi-Huqf burial from Immenhauser et al. [40] and Sattler et al. [51]. ^(b) Expected temperature of the samples on maximum burial depth: the geothermal gradient for Haushi-Huqf were obtained from Bergmann et al. [37]; the offshore-Arabian Gulf from Ehrenberg et al. [55]; Musandam Peninsula from Tibet-1ST well in Ali et al. [61]; and Oman Mountain from NB-22 well in Schütz et al. [62]. ^(c) The calculated temperature from Δ_{47} value using Anderson et al.'s [83] equation. ^(d) Difference (disparity) between max. clumped isotope temperature recorded on dolomite and calcite. ^(e) Including clumped isotope results of calcite sample measured by Patel [89]. ^(f) Including clumped isotope results of dolomite measured by Sena et al. [63].

For context, the results are compared to other studies in the Central Oman High [37] and the Sichuan Basin, China [114] (Figure 7). A study on co-located and coeval Permian dolomites and calcite in Central Oman High showed that the Δ_{47} values between the two minerals are similar [37]. This similarity can be attributed to (i) the dissemination of small rhombohedral dolomite found in the micrite matrix of the Permian limestone [37], which obscures the actual Δ_{47} of the calcite, or (ii) the lack of Δ_{47} reordering, because the strata were not buried deep enough to attain the high temperatures required (Figure 7). In contrast to our study, a study in the Sichuan Basin showed that the Δ_{47} temperatures of calcite are higher than co-located and coeval dolomites [114]. In this study, the dolomite and calcite were collected from the same rock specimen. To separate the minerals, the dolomite was isolated from calcite using an iterative chemical leaching process, which selectively dissolved calcite, leaving the dolomite in the residual sample [114,115]. However, such cleaning methods have the potential to cause alteration to the geochemical signal, mainly Mg/Ca and $\delta^{18}\text{O}$ [116]. Hence, the artificial alteration of the clumped isotope values after a repeated sample treatment cannot be ruled out, as most of the pre-treatments involving liquid [116] and plasma treatment [74] have previously shown a potential alteration.

Furthermore, the calcite and dolomites used in the Sichuan Basin study were collected from the same rock matrix [114], thus raising the question of the isotopic interaction between minerals during burial and recrystallisation (dissolution and precipitation). Thus, the acquisition timing for the Δ_{47} values of these minerals is uncertain, as the isotopic exchange between calcite and dolomite was not explained in the study. In comparison, co-located and coeval calcite and dolomites in the current study were collected in co-hosted but distinct beds of either pure calcite or dolomite to ensure no isotopic exchange between calcite and dolomite occurred and the value difference can be attributed solely to C-O bond reordering kinetics (Figure 7).

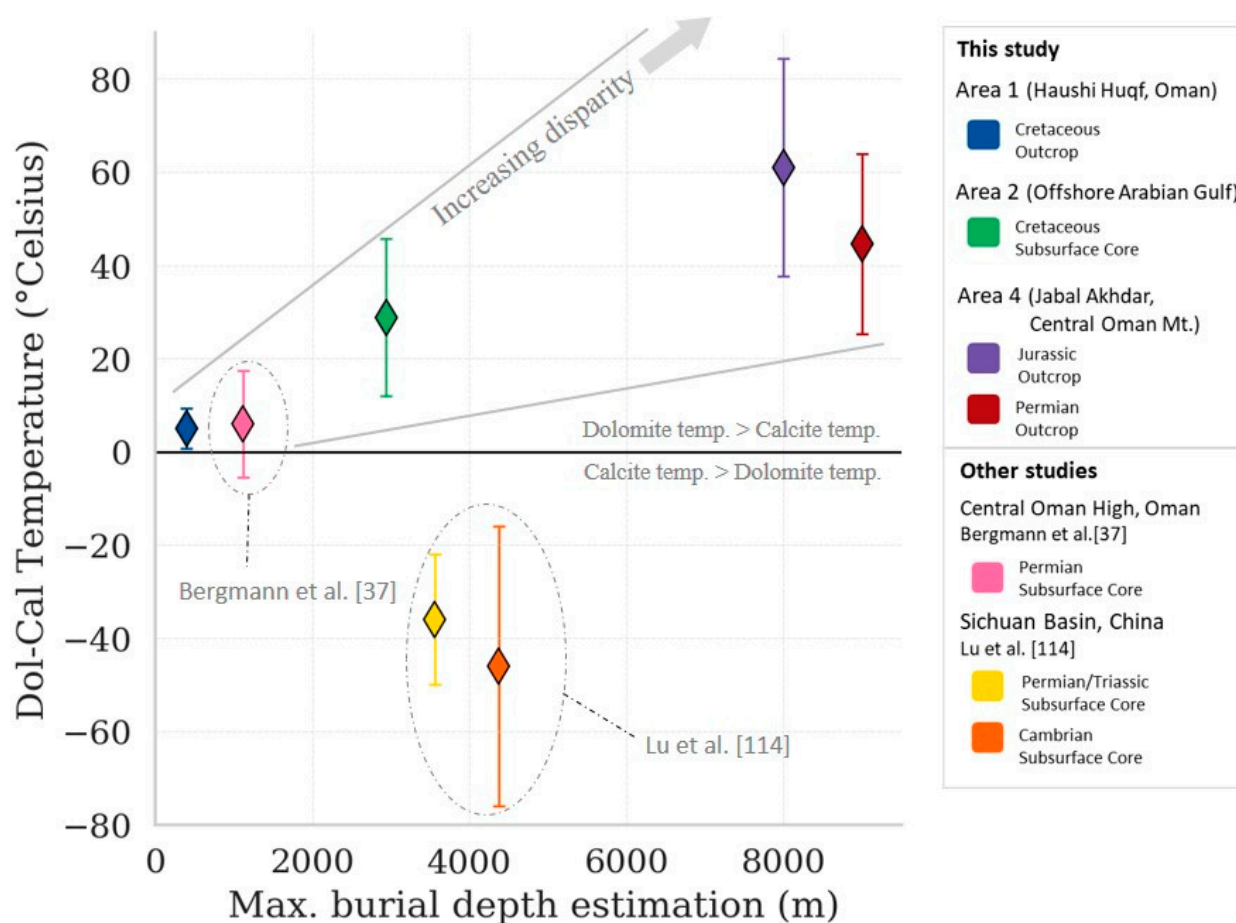


Figure 7. Difference between maximum Δ_{47} temperature recorded in dolomite and calcite with respect to its maximum burial reached (see Table 3). Published temperature difference from Bergmann et al. [37], and Lu et al. [114] are also shown. The disparity between temperatures recorded in both minerals increases as the rock formation is buried deeper.

5.4. Temperature and Dolomite Mineralogy Relationship

The mineralogy of dolomite has been used as a potential proxy to investigate the diagenetic processes and environment [117–119]. One of the fundamental mineralogical parameters examined in this investigation is cation ordering, which refers to the degree to which Mg and Ca cations are distributed in their cation layers in the dolomite lattice [69,120].

Laboratory experiments have shown that cation ordering develops at a faster rate at higher temperatures [69]. Data from global Phanerozoic dolomites also show a strong correlation between cation ordering and geological age and burial depth [121,122]. Here, we show that the early dolomite with a higher blocking temperature has a higher cation ordering compared to the early dolomites that record lower temperatures (Figure 8). In the Jurassic and Permian intervals, the lower blocking temperature appears to correspond to lower cation ordering, providing the empirical relationships between the two parameters (Figure 8). Our findings, therefore, support the hypothesis that the progressive development of cation ordering can be driven by temperature elevation, as previously suggested [69]. The findings also imply that observed differences in dolomite Δ_{47} blocking temperatures may be driven, at least in part, by the development of cation ordering.

The data presented here, however, do not indicate an interrelationship between stoichiometry and cation ordering (Table 2). The lack of correlation here implies that the Mg and Ca cations continue to organise in the lattice during recrystallisation, while the amount of Mg is most likely fixed by the composition of the initial dolomite and does not change significantly during temperature-driven recrystallisation in the closed-system.

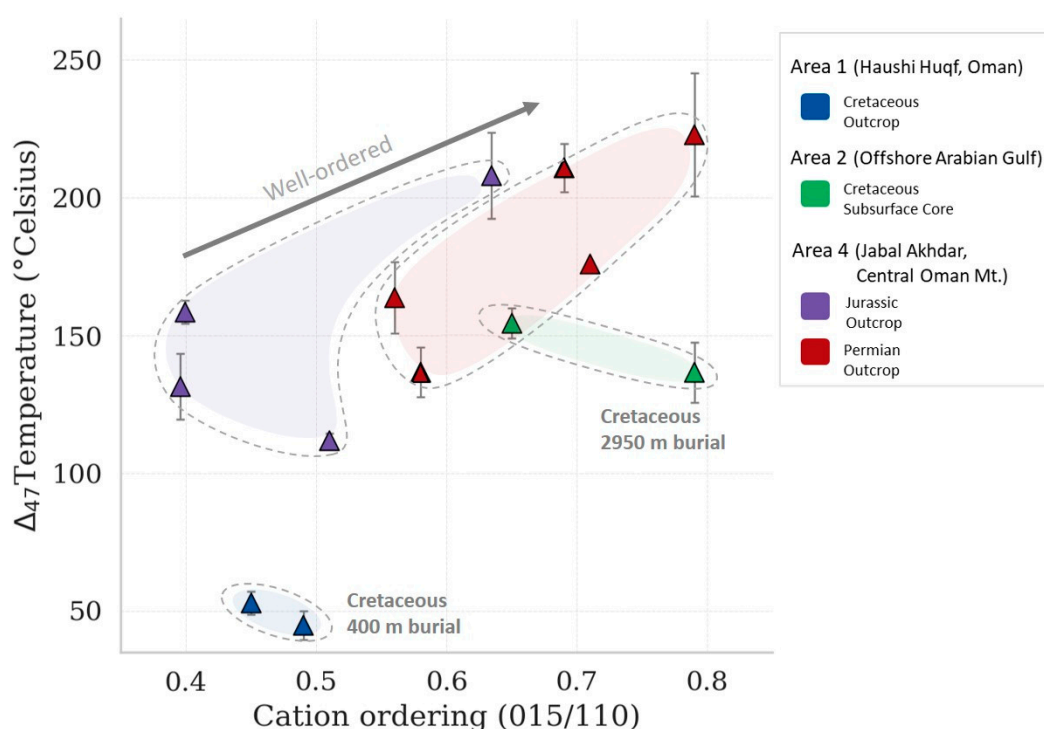


Figure 8. Dolomite {015}:{110} cation ordering and Δ_{47} temperature relationship. Cation ordering correlates positively with Δ_{47} temperature in two of the largest datasets, showing that high-temperature (low Δ_{47}) dolomite corresponds to well-ordered dolomite.

5.5. Modelling Applications

5.5.1. Offshore Arabian Gulf

In the Arabian Gulf area, the Δ_{47} temperatures of the calcites from the Cretaceous interval are consistent with those expected from the geothermal gradient (Table 3). The maximum Δ_{47} temperature recorded by calcite (126 °C) is less than one standard error away from the current temperature [55]. These results could suggest that this calcite records the temperature of maximum burial, and underwent only burial processes with no subsequent exhumation. However, the maximum Δ_{47} temperatures recorded in dolomite in the same interval (~7 m vertical difference) are inconsistent with the formation temperature of 136–154 °C. Our measurements show that they are 6–24 °C higher, suggesting the possibility of higher temperature history predating the current conditions. The recalculated $\delta^{18}\text{O}_{\text{fluid}}$ values show that all of our samples are formed in low water–rock ratios, fitting the isotope exchange reactions via solid-state clumped isotope bond reordering [7,8,18,20,21]. Thus, it is ideal for the forward modelling to test which process is accountable for the samples' Δ_{47} values, between low-water rock recrystallisation and solid-state clumped isotope bond reordering.

Our forward model results show that solid-state reordering during progressive burial in the Arabian Gulf is unlikely and does not fit the measured maximum temperatures recorded in calcite or dolomite (Figure 9A). In this model (peak 130 °C with 0.81 °C/Ma heating rate), the dolomite retained its initial Δ_{47} but calcite did not. The unaffected dolomite Δ_{47} was expected, as previous studies suggest that the rates of solid-state clumped isotope reordering are slower in dolomite than in calcite [16,22,123,124]. This model demonstrates that solid-state reordering by itself is unable to bring both minerals to their (present-day) equilibrium temperature.

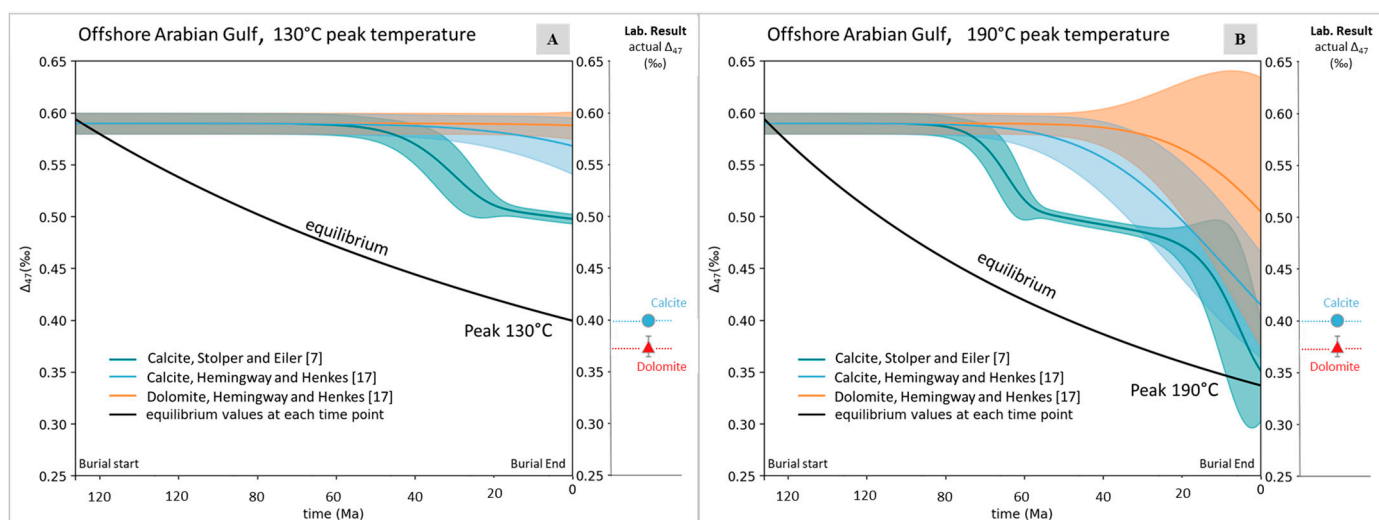


Figure 9. Result of forward model (A) with peak burial at present-day formation temperature of 130 °C, where the prediction and actual clumped isotope temperature are inaccurate. Forward model (B) with peak burial scenario of 190 °C, where the model hardly fits the prediction. The results of the Stolper and Eiler [7] equation on calcite minerals and the Hemingway and Henkes [17] equation on both calcite and dolomite are shown.

To reproduce the measured temperature of our samples, the peak temperature of the solid-state reordering heating model needed to be increased and was adjusted to 190 °C with a 1.32 °C/Ma heating rate. Based on the disordered kinetic model equation [17], the Δ_{47} for calcite predicted by this model reached 0.416 ± 0.050 ‰ (1SE), which is higher than the actual maximum temperature record of 0.400 ± 0.007 ‰ (1SE). Similarly, the predicted Δ_{47} dolomite in the model reached 0.502 ± 0.133 ‰ (1SE), which is also higher than the actual maximum temperature record of 0.368 ± 0.005 ‰ (1SE). The predicted calcite using the paired-diffusion model equation reached 0.353 ± 0.053 ‰ (1SE). The calcite mineral temperature prediction appeared to fit with the calibration considering the standard error margin (1SE), while the measured dolomite Δ_{47} temperature is at the very end of the wide error range for dolomite prediction (Figure 9B).

Using the results from the adjusted 190 °C peak model instead of the 130 °C peak model, we can argue that the Cretaceous interval in the offshore Arabian Gulf was likely buried ~1750 m deeper than its present-day depth to approximately 4700 m. In this scenario, the dolomite records a temperature of 154 °C and retains it, whereas the calcite cools to the present-day formation temperature of 126 °C. However, published burial histories in this area revealed no exhumation more significant than 200 m [55], meaning the Cretaceous interval is likely buried only ~200 m deeper than its present-day depth.

Given that our models suggest that the solid-state reordering process alone is unable to explain the current mineral temperature records, it is reasonable to postulate that both minerals likely underwent progressive recrystallisation during burial under low water–rock ratio conditions. In this case, there are two competing explanations for the observed difference in calcite and dolomite temperature records.

First, it is possible that the calcite in this interval records the current ambient temperature, while the dolomite records the older ambient temperature when the gradient geothermal was higher. This would mean that the temperature discrepancy between dolomite and calcite is a function of cooling in the geothermal gradient. It has been previously suggested that rifting in the basin was associated with an instantaneous increase in heat flow in response to the thinning of the lithosphere [125,126]. Other studies, however, suggest that a delay in increased heat flow after rifting was initiated [126–128]. Given that heat flow is a function of the geothermal gradient and thermal conductivity [129], we

argue that the geothermal gradient in the area was probably higher than that recorded in the dolomite.

Second, considering a minimum temperature difference of 6 °C between the coolest dolomite recorded in our samples and the current formation temperature, it is reasonable that the formation has experienced a minimum uplift of 200 m. This suggests that the difference in calcite and dolomite Δ_{47} temperature records reflects tectonic uplift, where the maximum burial temperature is only retained by dolomite. Either way, the data suggest that the temperature of this Barremian–Aptian interval of the Kharaib Formation was higher than present-day conditions.

5.5.2. Jabal Akhdar, Central Oman Mountains

In Jabal Akhdar, a burial history model by Grobe et al. [39] showed that the maximum burial depth reached by the Jurassic strata was ~8 km. The stratigraphic thickness between the Jurassic and the Permian samples is ~1 km, suggesting that the maximum burial depth reached by the Permian sample was ~9 km. Using information from the geothermal gradient from well NB-22 [62], the Jurassic sample should have reached 169 °C at a maximum burial depth of 8 km, while the Permian sample reached 187 °C at 9 km (Table 3). Interestingly, Δ_{47} data indicate that the maximum temperature recorded by Jurassic calcite is 147 ± 12 °C, similar to the predicted temperature at 8 km considering the standard error (SE) of the measurement (Table 3). The maximum Δ_{47} temperature recorded by Permian calcite is 178 ± 8 °C, which is also similar to the estimate at 9 km (Table 3). Coeval with the calcite, however, the maximum Δ_{47} temperature recorded by the dolomite in the Jurassic interval is higher (208 ± 15 °C), while the dolomite in the Permian interval recorded 223 ± 22 °C.

The Δ_{47} values measured for calcite and dolomite in Jabal Akhdar do not represent maximum burial temperatures but the blocking, or apparent equilibrium temperatures, which are sensitive only to the cooling rate [7,8,22]. Therefore, the maximum burial temperature reached by our Jurassic and Permian intervals should be higher, reflecting deeper burial. To test this, the exhumation model was created (Figure 10).

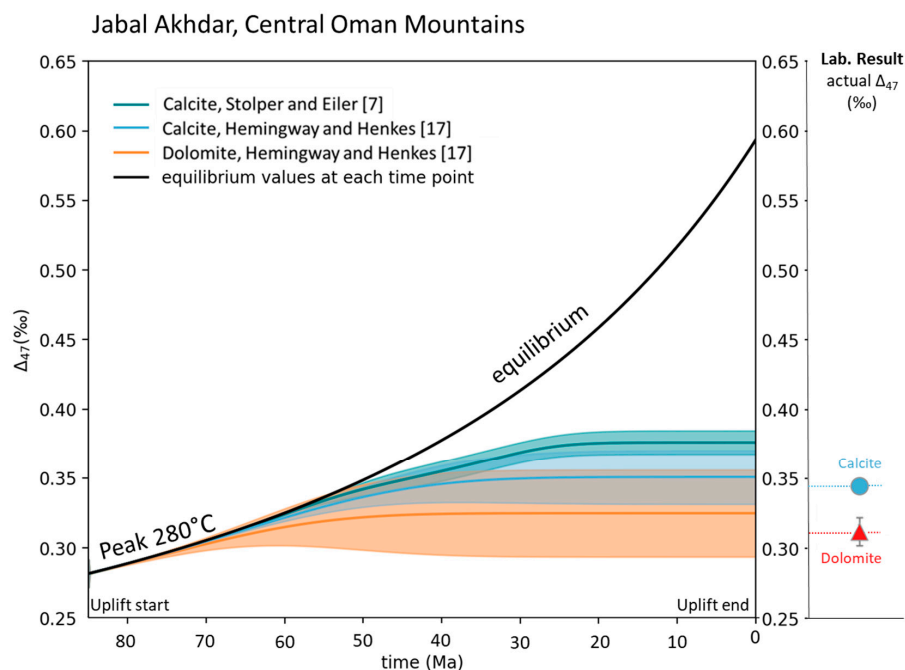


Figure 10. Results of the forward model from peak burial 280 °C cooled down to 25 °C. Using the Hemingway and Henkes model [17], the prediction fits the actual clumped isotope temperature. The result of the Stolper and Eiler [7] equation is also shown.

The exhumation model results show that the blocking temperature reached during the cooling/uplift event in Jabal Akhdar fits the measured maximum Δ_{47} temperature for both calcite and dolomite (Figure 10). To achieve this result, an initial peak temperature of 280 °C with a cooling rate of 3.18 °C/Ma was required. Only at this peak temperature did the calcite mineral model reach a Δ_{47} of $0.350 \pm 0.020\%$ (1SE), matching the actual (clumped isotope) measurement record of $0.346 \pm 0.001\%$ (1SE). Likewise, the end result of the dolomite mineral model reached a Δ_{47} of $0.325 \pm 0.031\%$ (1SE), matching the actual measurement record of $0.313 \pm 0.014\%$ (1SE).

These results contradict previous findings from illite–smectite analysis, where Aldega et al. [35] argued for peak temperatures of 150–200 °C on the northern flank and 120–150 °C on the southern flank of Jabal Akhdar. However, our predicted peak temperature of 280 °C is in accordance with findings reported from a zircon fission track study in the pre-Permian basement indicating peak temperatures up to 280 °C [130] and a Raman spectroscopy study on carbonaceous material indicating peak burial temperatures of 266 to 300 °C for the entire Jabal Akhdar [39,131].

The above case studies show that the Δ_{47} values of two co-located and coeval carbonate minerals can predict and confirm existing geological scenarios (i.e., Jabal Akhdar) and reveal a new scenario (i.e., Arabian Gulf).

6. Conclusions

New data from five sites in Oman demonstrate that co-located calcites and dolomites with the same thermal history record different clumped isotope values. The maximum clumped isotope temperatures recorded in the early dolomites are consistently higher than for co-located and coeval calcites. We suggest that this disparity is related to the difference in the kinetic parameters between the two minerals. Moreover, the difference between the calcite and dolomite Δ_{47} temperature appears to systematically increase with deeper burial, an observation that confirms previous laboratory experiments suggesting that the dolomite Δ_{47} records are more resistant to alteration during burial and exhumation.

Given that calcite is the most common mineral used in carbonate clumped isotope studies to reconstruct burial history, the results presented here suggest that, although the Δ_{47} temperature records of calcite in subsurface strata may appear to be in line with the gradient geothermal temperature, they may not convey the whole geological story. In the case of the Oman carbonates, a parallel examination of calcite and dolomite allows the identification of a previously undetected uplifting event in the offshore Arabian Gulf. By contrast, the calcite and dolomite records are able to accurately predict the peak temperature reached by the exhumed area of Jabal Akhdar. This study demonstrates the benefits of reconstructing thermal history using co-occurring calcite and early dolomite, and confirms that dolomite has a higher closing temperature during cooling and offers additional constraints on the burial model. Lastly, mineralogical data from the early dolomites are consistent with the hypothesis that the progressive development of cation ordering can be driven by temperature elevation, as previously suggested as a result of laboratory investigations.

Author Contributions: Conceptualization, Q.A. and C.M.J.; methodology, Q.A., S.E.K. and C.M.J.; modeling, Q.A.; validation, Q.A., S.E.K. and C.M.J.; investigation, Q.A., S.E.K. and C.M.J.; writing—original draft preparation, Q.A.; writing—review and editing, Q.A., S.E.K. and C.M.J.; visualization, Q.A.; funding acquisition, Q.A. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Indonesia Endowment Fund for Education.

Data Availability Statement: Data are contained within the article.

Acknowledgments: We are thankful to all members of the Carbonate Group, Imperial College London, for their help in the lab and constructive discussions to improve this paper, resulting its current form.

Conflicts of Interest: The authors declare no conflict of interest.

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