Rolling window regression of $\delta^{13}$C and $\delta^{18}$O values in carbonate sediments: Implications for source and diagenesis

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Abstract
Diagenetic alteration can produce, modify or erase significant biogeochemical information recorded in carbon and oxygen ($\delta^{13}$C and $\delta^{18}$O) values of marine carbonates throughout geological time. Understanding the type and extent of alteration experienced by a carbonate deposit can improve sedimentological and geochemical interpretations of events in Earth history. In this study, we present a new application of a statistical approach to aid in the identification and interpretation of sedimentological surfaces in a shallow marine carbonate sequence using a rolling window regression (RWR) analysis. RWR analysis evaluates the degree of covariation between two records and how it changes through time. Geological application of this statistical technique permits a new perspective on the fine scale variability in carbon and oxygen isotope records and the processes that generate them and provides a complementary tool for sedimentological interpretations. In this study, we apply RWR to $\delta^{13}$C and $\delta^{18}$O values from the Clino drilled into the western margin of Great Bahama Bank, a core that has been extensively altered by diagenetic processes, within the vadose, phreatic and marine burial zones. This core penetrates ~676 m of shallow marine to deeper slope deposits and contains a variety of both sedimentological and diagenetic events, including facies transitions, subaerial exposure surfaces, marine hardgrounds, firmgrounds and periods of reduced sedimentation rate. Using more than 1,200 analyses of paired $\delta^{13}$C and $\delta^{18}$O values, we have applied a RWR analysis to evaluate how the correlation between these two proxies changes at scales of 10, 30 and 100 m. The results of this study highlight the dynamic evolution of correlations between $\delta^{13}$C and $\delta^{18}$O values within diagenetic zones and in association with diagenetic surfaces and provide context for interpreting covariance in $\delta^{13}$C and $\delta^{18}$O values from the geological record.

Keywords
carbon and oxygen isotopes, Clino, diagenesis, Great Bahama Bank, sedimentological surface
Carbone rocks represent valuable archives of Earth’s biogeochemical cycles that record significant physical, chemical and biological events produced by evolution of new organisms, tectonic events, palaeoclimatic and extinction events, as well as periods of intense environmental change. The carbon and oxygen isotope composition of carbonates is of particular interest, with the δ18O providing information on temperature and salinity (Emiliani, 1955; Shackleton, 1967; Weber, 1964) while the δ13C values of marine carbonates are interpreted as a proxy for changes in plant evolution and physiology and the global carbon cycle through geological time (Veizer, Holser, and Wilgus, 1980; Shackleton and Hall, 1985; Hayes, Strauss, and Kaufman, 1999; Veizer et al., 1999; Bekker et al., 2008; Swanson-Hysell et al., 2010). These signals are often influenced by diagenetic changes during periods of sub-aerial exposure (Allan and Matthews, 1982; Christ et al., 2012; Gross, 1964; Gross and Tracy, 1966; Land, 1967) but can also be prevalent in the submarine realm where δ13C and δ18O values can be altered during marine burial diagenesis (Marshall and Ashton, 1980; Frank, Arthur, and Dean, 1999; Immenhauser, Creusen, Esteban, and Vonhof, 2000; Mutti and Bernoulli, 2003; Sattler, Immenhauser, Hillgärtner, and Mateu, 2005; Dickson, Wood, Al Rougha, and Shebl, 2008). However, unravelling the original signature of such events from post-depositional changes experienced by carbonate sediments can prove difficult. For example, although shallow water carbonate successions are frequently studied as a result of their widespread occurrence in the geological record, alteration by subaerial and marine diagenetic processes environments can change the original δ13C and δ18O values.

Previous workers have described specific and systematic changes in δ13C and δ18O values in carbonates affected by diagenetic alteration and changes in sediment source through time. Subaerial exposure surfaces are recognized by positive δ13C values derived from the oxidation of organic material and can be accompanied by more positive δ18O values as a result of evaporation in tropical conditions (Allan and Matthews, 1982; Beier, 1987; Joachimski, 1994; Immenhauser et al., 2002), while arid conditions inhibit the formation of significant δ13C and δ18O shifts (Christ et al., 2012). Distinct carbon and oxygen isotope signatures, including a positive covariance between δ13C and δ18O values, have been attributed to mixing zone alteration (Humphrey, 1988; Melim, Swart, and Eberli, 2004) and recently, diagenesis in the phreatic zone (Swart and Oehlerl, 2018). Marine burial diagenesis is generally characterized by more positive δ13C and δ18O values compared to meteoric zone alteration, with isotopic compositions that can approach the composition of the original platform-derived material (Killingley, 1983; Czerniakowski, Lohmann, and Wilson, 1984; Scholle and Halley, 1989; Derry, 2010). However, diagenetic alteration related to hardground formation in the marine burial zone has been proposed to shift δ13C values towards more positive values as a result of the precipitation of inorganic cement (Marshall and Ashton, 1980), as well as towards more negative δ13C values as a result of the oxidation of organic material during marine burial diagenesis (Swart and Melim, 2000; Dickson et al., 2008). Table 1 summarizes the characteristic carbon and oxygen isotope signatures associated with various diagenetic processes in carbonate environments. As a result of these characteristic signatures of diagenesis, some studies have proposed that the lack of such observed correlation between δ13C and δ18O values proves the original, unaltered nature of the deposit (Fike, Grotzinger, Pratt, and Summons, 2006; Grotzinger, Fike, and Fischer, 2011; Rose et al., 2012).

Changes in sediment source through time can also produce paired shifts in δ13C and δ18O values; positive shifts in both carbon and oxygen isotope records can be generated by shifting to dominantly platform-derived sediments, whereas a transition to more pelagic background sediments during periods of reduced sedimentation (sensu Kenter, Ginsburg, and Troelstra, 2001) can produce negative shifts in both δ13C and δ18O values from bulk carbonate sediments in the Bahamas (Swart and Eberli, 2005; Swart, 2008).

In order to address the need to separate high-quality records of biogeochemical events in Earth history from those that have been diagenetically altered beyond recognition from the original δ13C and δ18O values, many workers have developed strategic diagenetic screening systems that incorporates more geochemical aspects than just an analysis of the covariation of δ13C and δ18O values. These techniques include evaluating trace element concentrations and ratios of diagenetically relevant elements (Brand and Veizer, 1980; Banner, 1995; Frank, Lohmann, and Meyers, 1996; Metzger and Fike, 2013) and have been applied to carbonate δ13C and δ18O values to eliminate diagenetically altered samples from datasets to improve interpretation of original biogeochemical changes. Other workers have applied statistical techniques such as parametric and non-parametric statistical models (Oehlerl et al., 2012; Krissansen-Totton, Buick, and Catling, 2015), hierarchical cluster analysis (Coimbra, Marques, and Olóriz, 2019) and principal component analysis (Elek, 1988; Kazmierczuk and Jarzyna, 2006; Ma, 2011; Enikanselu and Ojo, 2012; Coimbra et al., 2017; among others) to datasets of elemental and carbon and oxygen isotope stratigraphy in ancient carbonates in order to quantify changes in sediment source, lithology, facies and diagenesis. In many cases, petrographic analysis of carbonate sediments and their cements, as well as core descriptions of sedimentologically relevant surfaces are often incorporated to prevent the interpretation of diagenetically altered samples as pristine records of original changes in the global carbon cycle.
In this study, we suggest that the application of rolling window regression (RWR) analysis can provide key insights in the identification of diagenetic alteration and its causal mechanisms in paired $\delta^{13}$C and $\delta^{18}$O values of marine carbonates. Recent geological studies have employed RWR analysis to datasets to determine how the degree of covariation between two datasets changes through time in palaeoclimatic studies with success (Moore, Grinsted, and Jevrejeva, 2008; Wang et al., 2014). The benefit of RWR analyses is that they highlight periods of strong positive and strong negative covariance between two datasets. We apply this method to assess the correlation between $\delta^{13}$C and $\delta^{18}$O values within a window of specific depths in the core. The window is then incremented by one unit and the covariance calculated again. A RWR of records of $\delta^{13}$C and $\delta^{18}$O values from carbonate deposits therefore provides insight into how their correlation evolves within and across diagenetic zones. As identification of sedimentological surfaces like subaerial exposures, marine hardgrounds and periods of reduced sedimentation rate can be problematic and subject to interpretation. RWR analysis can provide enhanced ability to target zones of potential alteration and thus cryptic sedimentological surfaces. In this case study, we have applied RWR analysis to a core from the Bahamas (Figure 1), to assess the potential of this technique to identify diagenetic zones, exposure surfaces, hardgrounds and firmgrounds in the geological record. Importantly, integrating sedimentological and diagenetic datasets has been

| Sedimentological interpretation | Carbon and oxygen isotope signatures | References |
|---------------------------------|--------------------------------------|------------|
| Calcrite                        | Negative shift in $\delta^{13}$C values and occasional positive shift in $\delta^{18}$O values | Allan and Matthews (1982) |
| Vadose                          | Variable and negative $\delta^{13}$C values; $\delta^{18}$O values homogeneous and negative | Allan and Matthews (1982) |
| Freshwater phreatic             | $\delta^{13}$C and $\delta^{18}$O values covary as sediments experience progressively less freshwater alteration with depth | Swart and Oehlert (2018) |
| Mixing zone                     | Principally a zone of dissolution, original $\delta^{13}$C and $\delta^{18}$O values expected | Swart and Oehlert (2018) |
| Marine burial diagenesis        | $\delta^{13}$C and $\delta^{18}$O values are relatively positive and reflect original composition | Allan and Matthews (1982) |
| Hardground (Type I)             | $\delta^{13}$C and $\delta^{18}$O values shift towards more positive values as a result of inorganic cement precipitation | Marshall and Ashton (1980) |
| Hardground (Type II)            | $\delta^{13}$C and $\delta^{18}$O values shift towards more negative values as a result of oxidation of organic matter during marine burial diagenesis | Swart and Melim (2000); Dickson et al. (2008) |

**TABLE 1** Summarizes the characteristic isotope signatures associated with various diagenetic processes in carbonate environments.

**FIGURE 1** Location of Clino core on western margin of Great Bahama Bank. Clino was drilled in 1991 during the Bahamas Drilling Project (Ginsburg, 2001)
shown to produce the most well-constrained interpretation of biogeochemical events (Grotzinger & James, 2000), and the results of this study suggest that RWR analysis is an additional method that can be used to improve interpretation of discontinuity surfaces and diagenetic alteration in shallow marine carbonates through geological time.

2 | MATERIALS AND METHODS

2.1 | Materials

In 1991, two continuous cores were drilled in the shallow water of western Great Bahama Bank (Ginsburg, 2001). These cores were drilled to test the interpretation of Eberli and Ginsburg (1987) that the Great Bahama Bank was composed of a number of smaller platforms which had coalesced together by progradation as well as to examine the diagenetic behaviour of sediments over the past 2–5 Myr. One of the cores collected by the Clino (Figure 1) is the focus of this study because it has proven to hold a range of both depositional and diagenetic information, making it an ideal candidate to test the ability of RWR to capture and identify a variety of diagenetic signatures and sedimentological surfaces. Furthermore, the system is well-constrained by previous workers who have investigated the carbon isotope composition of the original sediment and organic matter produced on the platform top (Swart, Reijmer, and Otto, 2009; Oehlert et al., 2012), the sedimentology and facies transitions (Betzler, Reijmer, Bernetà, Eberli, and Anselmetti, 1999; Eberli, 2000; Ginsburg, 2001; Kenter et al., 2001; Manfrino and Ginsburg, 2001; McNeill, Eberli, Lidz, Swart, and Kenter, 2001 Swart and Melim, 2000) and the various diagenetic events (Melim, Swart, and Maliva, 1995, 2001; Melim, Westphal, Swart, Eberli, and Munnecke, 2002; Swart and Kennedy, 2011; Oehlert and Swart, 2014; Swart and Oehlert, 2018) experienced by the sediments in this core.

2.2 | Geochemical Methods

This study makes use of 1,279 analyses of $\delta^{13}$C and $\delta^{18}$O values from bulk carbonates collected from Clino. Of these, 637 measurements were presented in a study by Melim et al. (2002) and 464 by Oehlert and Swart (2014). One hundred and seventy-four new measurements of $\delta^{13}$C and $\delta^{18}$O values in Clino were analyzed in this study to enhance the sampling density of Clino especially in the vadose and phreatic zones of the core (Figure 2). The $\delta^{13}$C and $\delta^{18}$O values reported by Melim et al. (2002) are a fundamental component of this analysis because sedimentological surfaces were sampled for geochemical analysis, while Oehlert and Swart (2014) avoided sampling sedimentological surface in an effort to preserve limited core material. The integration of these three datasets produces an average sampling interval of 0.52 samples per metre. The $\delta^{13}$C and $\delta^{18}$O values from all three studies were analyzed using standard phosphoric acid dissolution in a common acid bath at 90°C (Swart, Burns, and Leder, 1991), and the CO$_2$ resulting from this reaction was analyzed on a Finnigan MAT 251 (ThermoFisher Scientific) housed in the Stable Isotope Laboratory at the University of Miami, RSMAS. Data are reported in the conventional notation and reported relative to Vienna Pee Dee Belemnite (V-PDB). The same internal standard calibrated to NBS-19 (National Bureau of Standards) was used in all studies. Errors on these analyses were <0.1‰ based on replicate analysis.

2.3 | Statistical Methods

In order to evaluate how the correlation between $\delta^{13}$C and $\delta^{18}$O values at Clino changes through time, an RWR analysis was conducted on the combined dataset. In order to eliminate uneven sampling biases in the data, the samples were interpolated to a standard sampling interval of 1 m using a rectangular interpolation method (Davis, 1973). The regression analysis was then performed using variable window sizes (100, 30 and 10 m) and used to assess the impact of RWR on the generation of diagenetic and sedimentologically relevant observations. The values of $R$ considered statistically significant for each window are shown in Table 3.

In order to more easily assess the direction of the shifts in the un-interpolated $\delta^{13}$C and $\delta^{18}$O values from Clino, long-term trends in the data were removed through a process known as prewhitening (Cook, 1985; Storch, 1995), see our prewhitened results in Figure 3). Prewhitening is a statistical method of ‘eliminating or reducing short-term stochastic persistence to enable detection of deterministic change’ (Razavi and Vogel, 2018). Prewhitening was conducted in Matlab using the Acycle 0.1.3 package (Li et al., 2019), and the r-LOESS routine was selected to subtract long-term trends. For example, records of both $\delta^{13}$C and $\delta^{18}$O values show a large change from negative values near the top of the core associated with freshwater diagenesis to more positive values associated with original carbon and oxygen isotope compositions of the sediments from the platform top. These changes, and others, have been described in detail by Melim et al. (2002) and Swart and Oehlert (2018). Removing these lower frequency trends allows for enhanced recognition of small perturbations which might be associated with finer scale diagenetic events. The benefit of the prewhitening filter in the Acycle 0.1.3 package (Li, Hinnov, and Kump, 2019) in this study is to highlight the residual variability in the records of $\delta^{13}$C and $\delta^{18}$O values from Clino (Figure 3), allowing for better constraints on the direction of changes in the $\delta^{13}$C and $\delta^{18}$O values of each record. In conjunction with the RWR analysis, prewhitened $\delta^{13}$C and $\delta^{18}$O values improve the interpretation of the range
of possible physical, chemical and biological processes that generate positive and negative correlation coefficients in carbonate environments.

## 3 | RESULTS

A depth plot of δ¹³C and δ¹⁸O values which incorporates new data as well as published datasets (Melim, Swart, and Maliva, 2001; Oehlert and Swart, 2014) is shown in Figure 2. Table 2 details the type of correlation associated with each sedimentological surface identified by Manfrino and Ginsburg (2001) and Kenter et al. (2001) for each window size.

### 3.1 | The 100 m Window

The RWR analysis on the 1 m interpolated dataset using a 100 m window revealed zones of significant positive correlation as well as zones of significant negative correlation between δ¹³C and δ¹⁸O values in Clino (Table 3; Figure 4). Using a 100 m window, the vadose and upper portion of the phreatic diagenetic zones is characterized by a significant positive correlation between δ¹³C and δ¹⁸O values (Figure 2). In contrast, the base of the freshwater phreatic diagenetic zone is characterized by significant negative correlation between δ¹³C and δ¹⁸O values, possibly associated with the marine hardground observed by Kenter et al. (2001) at 219.7 mbmp. Additional zones of significant negative correlation between δ¹³C and δ¹⁸O values are associated with sedimentological surfaces in the Marine Burial diagenetic zone, including marine hardgrounds associated with erosion (367 mbmp) and marine hardgrounds associated with reduced sedimentation rate (536.3 and 542 mbmp respectively). Significant positive correlations in the Marine Burial zone were also observed at 585.1 mbmp, in association
with a period of reduced sedimentation rate, as well as below 600 mbmp where reduced sedimentation rate and periods of minor erosion were observed (Kenter et al., 2001).

### 3.2 | The 30 m Window

The 30 m window RWR analysis on the 1 m interpolated dataset showed higher resolution variability in the
correlation between $\delta^{13}C$ and $\delta^{18}O$ values in Clino (Table 3; Figure 5) when compared to the results of the 100 m window analysis. Some zones in Clino exhibiting a high degree of both positive and negative correlation. Using a 30 m window, much of the upper vadose zone is not significantly correlated (Figure 5). Between ~90 and 150 mbmp, the $\delta^{13}C$ and $\delta^{18}O$ values are positively correlated, spanning the boundary between the lower vadose and uppermost phreatic diagenetic zones (Figure 5). A zone of significant positive correlation is observed at the base of the phreatic diagenetic zone around 210 mbmp where a marine hardground is observed. In the marine burial diagenetic zone, three significant zones of negative correlation were observed corresponding to marine hardgrounds and periods of reduced

| Statistical parameter | 10 m window | 30 m window | 100 m window |
|-----------------------|-------------|-------------|--------------|
| Degrees of freedom    | 8           | 28          | 98           |
| $p$-value             | .05         | .05         | .05          |
| Significance level $R$| ±0.632      | ±0.361      | ±0.197       |
| $p$-value             | 0.1         | —           | —            |
| Significance level $R$| ±0.549      | —           | —            |

**TABLE 3** Summary of statistical parameters used in RWR analysis for determination of significance

**FIGURE 3** Prewhitened $\delta^{13}C$ and $\delta^{18}O$ values from Clino. Diagenetic zones are labelled following convention presented in Swart and Oehlert (2018). Mineralogy is from Melim et al. (2002). Dunham classification and diagenetic surfaces in the Marine Burial Zone are from Kenter et al. (2001), while surfaces in the Vadose and Phreatic Diagenetic Zones are from Manfrino and Ginsburg (2001)
sedimentation rate. In contrast, three zones of significant positive correlation are observed in the marine burial diagenetic zone and are associated with periods of reduced sedimentation rate (Figure 5).

### 3.3 The 10 m Window

The RWR analysis of the δ¹³C and δ¹⁸O values from Clino using a 10 m window revealed much higher frequency variability in the R values. Both significant positive and negative correlations can be observed in this record. Near-significant and significant positive correlations are associated with subaerial exposure surfaces in the vadose zone (Table 3; Figure 6), while positive correlations in the phreatic zone appear to be associated with changes in facies as described by the Dunham Classification (from Kenter et al., 2001). One significant negative correlation was observed in the phreatic diagenetic zone in association with a burrowed firmground at 197.4 mbmp, while a positive correlation was observed in association with a marine hardground within the phreatic zone (219.7 mbmp, Figure 6). Within the marine burial diagenetic zone, eight zones of significant positive correlation were observed, while only three zones of negative correlation were observed (Figure 6). Most of the zones of positive correlation occurred in between of sedimentological surfaces, except for the period of reduced sedimentation rate observed at 379 mbmp (Figure 6). Zones of significant negative correlation were observed to occur in association with marine hardgrounds (Figure 6).
DISCUSSION

With 1,278 measurements of δ¹³C and δ¹⁸O values from ~670 m of core, this Clino dataset provides a high-resolution perspective into variability in carbon and oxygen isotope composition resulting from diagenetic alteration and facies changes, and how these features are manifested at diagenetic surfaces. The incorporation of statistical analysis (summarized in Table 3) of the change in the correlation coefficient, R, between δ¹³C and δ¹⁸O values using a moving window throughout the core permits new insights into the stratigraphic heterogeneity and diagenetic zonation of shallow marine carbonate environments after being subjected to meteoric and marine burial diagenesis.

4.1 | Diagenetic zones highlighted by longer window lengths

The 100 m window encompasses nearly the entire section of the core impacted by alteration within the freshwater phreatic zone (Figure 4, 100–180 mmbp). The upper portions of the freshwater phreatic zone driven by particularly aggressive dissolution close to the water table as organic matter is oxidized (Budd, 1988; McClain, Swart, and Vacher, 1992, 1994; Swart and Oehlert, 2018) are expressed as a strong positive covariation between δ¹³C and δ¹⁸O values, supporting the interpretation that the rock has been diagenetically altered (Allan and Matthews, 1982). Comparing traditional covariation analysis which bin the carbon and oxygen isotope values by diagenetic zone,
similar trend is observed in the $R$ values. Previous workers evaluating the relationship between $\delta^{13}C$ and $\delta^{18}O$ values found $R$ values ~0.9 in the freshwater phreatic zone (Oehlert and Swart, 2014; Swart and Oehlert, 2018) and a similar result is found with the RWR (Figure 4). In addition to the expected positive correlation, the base of the freshwater phreatic zone exhibits a significant negative correlation between $\delta^{13}C$ and $\delta^{18}O$ values at around 220 mbmp (Figure 4), which occurs in association with a marine hardground described by Kenter et al. (2001).

Furthermore, using a 100 m window demonstrates that the peak correlation coefficient ($R > 0.8$) occurs between 130 and 150 mbmp, with decreasing significance of the $R$ value observed above (<130 mbmp) or below (>150 mbmp) this zone (Figure 4). This observation suggests that windows centred between 130 and 150 mbmp encompass the widest ranges in $\delta^{13}C$ and $\delta^{18}O$ values, likely generated by intense diagenetic alteration and recrystallization by meteoric fluids with $^{18}O$- and $^{13}C$-depleted isotopic compositions (Gross, 1964; Land, 1967; Allan and Matthews, 1982) that repeatedly influenced this section of the core during the Pleistocene (Swart and Oehlert, 2018).

The freshwater phreatic zone is not the only diagenetic zone to have more than one style of covariation between the $\delta^{13}C$ and $\delta^{18}O$ values. Previous analysis of the correlation between $\delta^{13}C$ and $\delta^{18}O$ values in the marine burial zone (180–700 m) of Clino suggest a weak negative correlation between $\delta^{13}C$ and $\delta^{18}O$ values (Oehlert and Swart, 2014; Swart and Oehlert, 2018). The potential for multiple different types of covariation within the
marine burial realm was elucidated using a 100 m window in this study (Figure 4). Broadly, there are three major intervals of strong negative correlation interjected by two intervals of strong positive correlation at depths greater than ~220 mbmp (Figure 4). The variations in the nature of the correlation between δ¹³C and δ¹⁸O values can be attributed to changes in facies, which likely reflects changes in sediment source during sea-level highstands and lowstands (Immenhauser, Della Porta, Kenter and Bahamonde, 2003; Kenter et al., 2001; Swart, 2008; Swart and Eberli, 2005), and may also reflect large-scale changes in diagenetic style associated with marine hardgrounds and periods of reduced sedimentation rate described by Kenter et al. (2001). Since δ¹³C and δ¹⁸O values are often rock buffered during marine burial diagenesis (Banner and Hanson, 1990; Derry, 2010) the range in δ¹³C and δ¹⁸O values resulting from marine burial diagenesis is low compared to the vadose or phreatic zones. Rock buffering therefore produces more subtle changes in δ¹³C and δ¹⁸O values and prevents the development of distinct endmembers in the marine burial diagenetic zone. Since the range of δ¹³C and δ¹⁸O values is so narrow, regressing the δ¹³C and δ¹⁸O values from the marine burial zone as a whole essentially superimposes these five zones of variable R, obscuring important sedimentological and diagenetic inferences (Figure 4). In contrast, the RWR analysis highlights five intervals with different correlations between δ¹³C and δ¹⁸O values (Figure 4), supporting interpretations of variation in sedimentation style or diagenetic alteration.

### 4.2 Diagenetic surfaces identified by shorter window lengths

One of the most significant changes in reducing the window size to 30 m can be observed in the top 20–60 mbmp in the vadose diagenetic zone (Figure 5). Compared to the 100 m window (Figure 4), the 30 m analysis suggests that much of the vadose zone does not exhibit a significant correlation between δ¹³C and δ¹⁸O values (Figure 5). This is consistent with the original interpretation of Allan and Matthews (1982) in which the δ¹³C values are described as being highly variable and negative, and co-occurring δ¹⁸O values are relatively stable at negative values as well.

### 4.3 Comparison of δ¹³C and δ¹⁸O shifts with RWR analysis

Comparison of shifts in the δ¹³C and δ¹⁸O values, RWR analysis and sedimentological surfaces as described in Manfrino...
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and Ginsburg (2001) and Kenter et al. (2001) provides insight into the depositional and diagenetic processes that should be considered when interpreting such geochemical records (Table 2).

**4.3.1 | Subaerial exposure surfaces**

Ten subaerial exposure surfaces were identified in the Clino core using sedimentological indicators of subaerial exposure such as laminations of the surface, induration, as well as the presence of a shell layer, blackened pebbles, lithoclasts, coated grains, leaching, vugs and facies changes (Manfrino & Ginsburg, 2001).

Comparing perturbations of the $\delta^{13}$C and $\delta^{18}$O values in the top 102 mbmp of Clino (Figure 7) to the significance thresholds of the RWR analysis suggests two different types of diagenetic alterations are recorded in the vadose zone. All 10 subaerial exposure surfaces are associated with more negative $\delta^{13}$C values. Six of these also have trends towards negative $\delta^{18}$O values below the surfaces (Table 2; Figure 7). The remaining four subaerial exposure surfaces are associated with trends towards negative $\delta^{13}$C and positive $\delta^{18}$O values (Table 2)—typical of the interpretation of Allan and Matthews (1982). It is possible that these four surfaces experienced relatively prolonged periods of exposure compared with other surfaces, which allowed for longer periods of evaporation and subsequent $^{18}$O enrichment of the rain water (Allan and Matthews, 1982). This interpretation is supported by higher concentrations of Al at many of the subaerial exposure surfaces (Manfrino & Ginsburg, 2001) presumably derived from greater dust input during longer periods of exposure (Table 2).

While some of these subaerial exposure surfaces exhibit significant negative relationships between $\delta^{13}$C and $\delta^{18}$O values in the 10 m RWR analysis (Figure 6), evaluation of the anomaly dataset presented in Table 2 suggests that a higher sampling resolution is required as the average depths between sampled intervals in the top ~100 mbmp (0.36 m), may not adequately resolve the full geochemical expression of the subaerial exposure surfaces. Furthermore, subaerial exposure surfaces forming in arid conditions are expected to express different geochemical behaviour when compared to surfaces forming in humid or tropical conditions (Allan and Matthews, 1982; Christ et al., 2012).

Published studies of the thickness of subaerial exposure surfaces in other tropical carbonate environments suggest a range between 0.5 and 3.0 cm in the Recent geological past of Barbados (James, 1972), between 5 and 35.0 cm in the Triassic Carbonate Platforms of Northern Italy (Hardie, Bosellini, and Goldhammer, 1986) and reaching thickness between 10 and 50 cm in the modern Barbados and the Mississippian of Kentucky (Harrison and Steinen, 1978). Crusts associated with subaerial exposure during the Pleistocene in the Florida Key were described between 1 and 6 cm thick by Multer and Hoffmeister (1968). Based on these measured subaerial exposure surfaces, it is likely that the resolution of our dataset only partially records the carbon and oxygen isotope signatures associated with the SE observed at Clino. Improving the density of the analyses could provide important insights into sedimentological and diagenetic processes that govern variable relationships between $\delta^{13}$C and $\delta^{18}$O values associated with subaerial exposure surfaces through time.

**4.3.2 | Periods of reduced sedimentation rate**

Periods of low sedimentation rate were associated with an average of four sedimentological indicators by Kenter et al. (2001), including the degree of lithification, sediment texture, abrupt change in mineralogy, changing abundances of planktonic foraminifera, TOC, insoluble residue and lithoclasts, as well as a change in gamma ray measurements.

The prewhitened $\delta^{13}$C and $\delta^{18}$O data (Figure 3) for periods of reduced sedimentation consist predominantly of two different categories; those with negative changes in $\delta^{13}$C values, and those with positive changes. Three of the five periods of reduced sedimentation were associated with negative changes in

| Table 4 | Hypothetical relationships between $\delta^{13}$C and $\delta^{18}$O shifts, RWR correlation coefficients, sedimentological and diagenetic processes and their interpretation in the geological record. Anomalies for diagenetic processes are defined in the downcore direction to reflect the direction of influence of post-depositional alteration, while anomalies related to sedimentological shifts are described in the upcore direction to reflect syndepositional sedimentation trends |
|---------|---------------------------------------------------------------|
| Interpretation | Sedimentology | Diagenesis | RWR correlation | $\delta^{13}$C shift | $\delta^{18}$O shift |
| Marine hardground | Sulphate reduction | Negative | $-$ | $+$ |
| Marine hardground | Marine cementation | Positive | $+$ | $+$ |
| Subaerial exposure | FW diagenesis | Positive | $-$ | $-$ |
| Subaerial exposure | FW diagenesis | Negative | $-$ | $+$ |
| Aggradation/low stand | Facies change | Positive | $-$ | $-$ |
| Progradation | Facies change | Positive | $+$ | $+$ |
δ¹³C values, while two were associated with positive changes in δ¹⁸O values (Table 2). However, two of the low sedimentation rate intervals with trends towards negative δ¹³C values were observed in close association with large depositional hiatuses at 367 and 536.3 mbmp, suggesting that they were overprinted by the significant diagenetic alteration occurring at these intervals (Figure 3; Table 2). Facies changes can be ruled out as a mechanism for generating variable correlations between the different periods of reduced sedimentation since they all occur in sediments described as skeletal–peloidal without noted facies transitions (Kenter et al., 2001; Figure 3). Small-scale variations in carbonate mineralogy from Melim et al. (2001) suggest varying degrees of diagenetic alteration in association with these surfaces, with dolomitization being the primary driver for alteration. Pliocene sea-level variability, although not as dramatic as its Pleistocene counterpart (Raymo, Lisiecki, and Nisancioglu, 2006), could explain variable rates of sedimentation on the upper slope of the Great Bahama Bank during this time period. Changes in the carbon and oxygen isotope composition and the significance of RWR analyses in these instances should be cautiously interpreted, since it is likely that they have been dramatically altered from original signatures caused by periods of reduced sedimentation to reflect diagenetic signatures associated with the development of marine hardgrounds.

Only two of the periods of reduced sedimentation rate produced significant correlations between δ¹³C and δ¹⁸O values in Clino (474 and 585.1 mbmp, Figure 3). Positive RWR correlations can be generated when δ¹³C and δ¹⁸O values covary through either positive or negative shifts, RWR correlations should be compared to prewhitened records of δ¹³C and δ¹⁸O values for accurate interpretation of sedimentological and diagenetic processes that generate the correlation. For example, the period of reduced sedimentation rate at 474 mbmp has a positive correlation in the RWR analysis (Figure 5) and the shifts in δ¹³C and δ¹⁸O values are both positive (Figure 3). Since this period of reduced sedimentation is associated with mainly composed of sediments containing low magnesium calcite and small amounts of dolomite, it is possible that this period of low sedimentation occurred during a sea-level highstand or progradational pulse, where more platform-derived materials were being deposited on the slope in a slower rate, producing a positive shift in δ¹³C and δ¹⁸O values. In contrast, the period of reduced sedimentation rate at 585.1 mbmp has a positive correlation in the RWR analysis (Figure 5), but the anomalies in δ¹³C and δ¹⁸O values are both negative (Figure 3), possibly suggesting an aggradational or lowstand phase of the platform, with higher contributions of pelagic materials deposited.

4.3.3 Marine hardgrounds

Based on 15 lithological criteria, five marine hardgrounds were identified by Kenter et al. (2001, see our Table 2). On average, marine hardgrounds had 5.6 of the 15 possible criteria for interpreting discontinuity surfaces framed by Kenter et al. (2001), with the predominant sedimentological indicators for marine hardgrounds being abrupt upcore decreases in cementation or lithification, abrupt changes in grain composition or sediment texture, abrupt changes in mineralogy and changes in the gamma ray record.

The δ¹³C and δ¹⁸O values revealed that hardgrounds are often characterized by changes to more negative δ¹³C and positive δ¹⁸O values (Figure 3), and they exhibit both positive and negative correlations with each other in the 30 m window RWR analysis (Figure 5). Two of the most significant hardground observations occur at 367 and 536.3 mbmp. The hardground at 367 mbmp is associated with the transition from skeletal to peloidal-dominated background sediments, where mixed peloidal–skeletal packstone are replaced by peloid-dominated chalky wackestones to packstones (Kenter et al., 2001). The cause of this facies change was interpreted as a change the platform architecture which is thought to have transitioned from a reef rimmed platform to a flat-topped platform (Beach and Ginsburg, 1980; Kenter et al., 2001) in the late Pliocene around 3.4 Mya (McNeill et al., 1988) associated with the first episode of post Miocene cooling (McDougall and Wensink, 1966; Prell, 1984; Shackleton, Hall, and Boersma, 1984). The change towards negative δ¹³C values likely results from the oxidation of organic matter during the period of non-deposition, while the positive changes in δ¹⁸O values likely occurs as carbonates are recrystallized at colder water temperatures at depth relative to the warmer shallow waters in which they were formed (Marshall and Ashton, 1980; Immenhauser et al., 2000; Swart and Melim, 2000; Mutti and Bernoulli, 2003; Sattler et al., 2005; Dickson et al., 2008). Peaks in dolomite concentration also occur in conjunction with this marine hardground, suggesting that sulphate reduction and dolomitization processes likely dominated the diagenetic environment (Swart and Melim, 2000). In addition, the marine hardground at 536.3 mbmp has been described to occur with a significant change in sediment composition dated to the late Miocene–early Pliocene transition (Kenter et al., 2001). This hardground shows the most extreme changes in the prewhitened records of δ¹³C and δ¹⁸O values, with a negative shift in δ¹³C values that is 2‰ more negative than the mean, and a positive δ¹⁸O shift that is more than 4‰ greater than the mean (Figure 3). This is consistent with a period of 2–4 Myr of non-deposition (McNeil et al., 2001) and enhanced rates of bacterial sulphate reduction and dolomitization at colder, deeper water depths.

Three marine hardgrounds observed between 200 and 300 mbmp do not show any significant correlations in the RWR analysis (Figure 5). Three possible explanations exist: (a) the carbon and oxygen isotope signature of these events is suppressed, suggesting the hiatuses were shorter in duration,
(b) the variations in sediment type that co-occur with the hardgrounds may obscure the signatures of the processes that formed the hardground or (c) the finer scale nature of these shorter hiatuses requires a higher sampling density and smaller window length to resolve statistically significant correlations between δ¹³C and δ¹⁸O values.

4.3.4 | Potential surfaces/zones

Evaluation of the results of the RWR analyses conducted in this study permits a generalization of the hypothetical relationships between δ¹³C and δ¹⁸O anomalies, RWR correlation coefficients, sedimentological and diagenetic processes and their interpretation in the geological record, which are presented in Table 4. Negative correlations observed in the RWR analysis can be generated by sulphate reduction associated with the formation of marine hardgrounds, as well as diagenetic alteration associated with some subaerial exposure surfaces. Both processes produce negative δ¹³C and positive δ¹⁸O anomalies, demonstrating the importance of incorporating sedimentological and sequence stratigraphic datasets with geochemical records from carbonate environments (Grotzinger & James, 2000). Positive RWR correlations with trends towards positive δ¹³C and δ¹⁸O values are observed to occur in association with marine cementation and sedimentological facies changes associated with margin progradation. Positive RWR correlations with trends towards negative δ¹³C and δ¹⁸O values occur in some subaerial exposure surfaces, and in conjunction with facies changes resulting from reduced contribution of sediments from the platform carbonate factory during periods of aggradation and/or sea-level low stands.

While it is well-established that Clino has experienced extensive diagenetic alteration (Ahm, Bjerrum, Blättler, Swart, and Higgins, 2018; Melim, Swart, and Maliva, 1995; Melim et al., 2001; Oehlert and Swart, 2014; Stewart, Gutjahr, Pearce, Swart, and Foster, 2015; Swart and Kennedy, 2011; Swart and Melim, 2000; Swart and Oehlert, 2018), the RWR analysis presented in this study highlights the possibility of previously unrecognized sedimentological changes, diagenetic surfaces or zones of diffuse diagenetic alteration. Fourteen possible surfaces, sedimentological transitions or diagenetic zones were identified by this analysis and are listed in bolded text in Table 2. Four of these are likely to be caused by facies changes associated with aggradation and/or low-stand sedimentation pathways, as evidenced by positive RWR correlations with trends towards negative δ¹³C and δ¹⁸O values (265.5, 573.5, 616.5 and 654.5 mbmp, Table 2). Three are possibly caused by marine hardground formation, with one forming as a result of sulphate reduction producing negative δ¹³C and positive δ¹⁸O anomalies (349.5 mbmp), while the other two have positive δ¹³C and δ¹⁸O anomalies, suggesting marine cementation associated with hard ground formation (243.5 and 516.5 mbmp). Subaerial exposures are likely drivers for the shallow surfaces identified in Table 2 (27.5, 106.5, 117.5, 131.5 and 156.5 mbmp). Without further examination of the core and higher resolution sampling analysis, it is difficult to tell if these surfaces are unique subaerial exposures or if they represent stacked or penetrative calcretes from a single subaerial exposure of the Great Bahama Bank platform. Although further sedimentological and geochemical analysis would be warranted to investigate each of these previously undocumented surfaces, the application of RWR analysis to paired δ¹³C and δ¹⁸O values permits a rapid assessment of possible diagenetic zones, sedimentological transitions and formation of significant surfaces. The RWR analysis may be particularly useful in ancient deposits where sedimentological transitions are difficult to discern as a result of the primary micritic nature of the deposit, or by extensive diagenetic alteration through time which can reduce or even erase evidence of lithological changes representing changes in the depositional environment.

5 | CONCLUSIONS

The results of this study demonstrate the ability to improve interpretations of diagenetic surfaces and zones using a RWR statistical analysis in deposits of shallow marine carbonates. Continuous downcore RWR analysis highlights diagenetic surfaces, zones of diagenetic alteration, varying degrees of lithification, changes in sediment source through time and the geochemical trends that characterize them. It is important to note, however, that the quality of insight into diagenetic processes and the depths at which they influence the δ¹³C and δ¹⁸O values of carbonates in such an analysis is related to the temporal resolution of the dataset. Subaerial exposure surfaces were resolved with an average spacing of 0.3 m between samples, but based on previously published studies, increased sampling resolution would likely provide more insights into diagenetic processes at the scale of subaerial exposure surfaces (0.5–50 cm). Consideration for the scale of the geochemical changes targeted should be incorporated into sampling strategies for both outcrop and core analyses. The results of this study suggest that an average spacing of 0.25–0.5 m between samples will provide insights into the variety of physical, chemical and biological processes occurring at different depths within a single diagenetic zone. Understanding the evolution of the correlation coefficient throughout these diagenetic zones can provide enhanced insight into the processes and the depths at which they operate in order to generate the observed anomalies in the δ¹³C and δ¹⁸O values of carbonate deposits. Importantly, positive correlations in the RWR analysis can be generated by both coupled positive or coupled negative shifts in δ¹³C and δ¹⁸O values, and accurate interpretation of the geochemical significance of such events requires a careful integration of sedimentological and diagenetic datasets. Comparison of
RWR analysis with trends in paired $\delta^{13}$C and $\delta^{18}$O values suggests that there are unique geochemical signatures of sulphate reduction, marine cementation, freshwater diagenesis and facies changes that can be used to enhance interpretations of geochemical records in shallow marine carbonate successions throughout geological time.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA SHARING

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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