



Shell weights of foraminifera trace atmospheric CO₂ from the Miocene to Pleistocene in the central Equatorial Indian Ocean

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The Maldives Sea is a region dominated by the South Asian monsoon (SAM) and at present, a CO₂ source to the atmosphere. Ti/Al elemental ratios from Site U1467 and U1468 recovered from the Maldives Sea show a gradual increase from ~12 Ma and indicate terrigenous inputs to this region associated with increasing wind intensity associated with initiation of the SAM. Shell weights of planktonic foraminifera, *Globigerinoides trilobus* have been used to understand variations in surface water carbonate ion concentration for the last 20 Ma. Shell weights show a good correspondence with global CO₂ records and show heavier shell weights during the colder periods than compared to warmer intervals which reveals that the Maldives Sea behaved similar to other tropical oceanic regions in terms of its surface water carbonate chemistry. A significant decrease in CaCO₃ wt.%, decrease in foraminifera shell weights and dissolution of spines along with an increase in organic carbon (OC%) towards 10.5 Ma is linked to the reduced carbonate deposition and increased productivity during monsoon which is a feature in all tropical sediment cores. Lower shell weights and dissolution features on foraminiferal shells were observed during periods of intense Oxygen Minimum Zone (OMZ) suggesting calcite dissolution due to an increase in bottom water CO₂.

Keywords. Maldives Sea; Equatorial Indian Ocean; shell weight; foraminifera; pCO₂; South Asian monsoon.

1. Introduction

The Neogene period was characterized by major changes in sea level and ocean circulation, along with fluctuations and intensification of the South Asian monsoon (SAM) at ~12.9 Ma, of which the Maldives carbonate edifice bears a tropical marine record (Betzler *et al.* 2016). The Neogene period experienced a warm period, the Miocene Climatic Optimum (MCO) which occurred in late Early

Miocene ~17 Ma and was associated with warming of mid-latitudes by 6°C than present (Flower and Kennett 1994), followed by a transition period, the middle Miocene Climate Transition (MMCT) which marks the period of climate cooling, increased ice sheets and lowering of global sea-level (Miller *et al.* 2005). The initiation of Northern Hemisphere glaciation (NHG) during late Pliocene (~2.8 Ma) was the last major climate transition in the Cenozoic (Maslin *et al.* 1998).

The phased uplift of Himalayan–Tibetan complex is said to be a major reason for the onset or intensification of the SAM (Zhisheng *et al.* 2001). Long records of the SAM from the Indian Ocean are few (Kroon *et al.* 1991; Gupta *et al.* 2004, 2015; Clift *et al.* 2008; Betzler *et al.* 2016, 2018; Tripathi *et al.* 2017; Bialik *et al.* 2019). Various studies have fixed the timing of monsoon intensification between 30 and 7 Ma (Ramstein *et al.* 1997; Fluteau *et al.* 1999; Gupta *et al.* 2004; Clift *et al.* 2008). Amongst the ongoing discussions on SAM initiation, Betzler *et al.* (2016) provided an age of 12.9 Ma for the onset of an abrupt SAM. Associated with the monsoons is upwelling, especially in coastal regions as seen in the Arabian Sea which makes the Arabian Sea a large CO₂ source to the atmosphere (Goyet *et al.* 1998; Sarma *et al.* 2003). Observations suggest that the Indian Ocean north of 10°S is a net source for atmospheric CO₂, i.e., positive sea–air CO₂ flux (Sarma *et al.* 2013). Though monsoon records as discussed are available, pCO₂ records from the Indian Ocean are rare (Foster *et al.* 2012) and are mainly from regions that have surface seawater CO₂ in equilibrium with the atmosphere. Increase in surface water pCO₂ leads to a decrease in surface water [CO₃²⁻], one of the important carbonate system parameter, thus decreasing the capacity of foraminifera to calcify and hence the lighter shells (Barker and Elderfield 2002). Shell weights of planktonic foraminifera have the potential to trace atmospheric CO₂ through resultant changes in [CO₃²⁻] of surface waters (Barker and Elderfield 2002; Naik *et al.* 2010), however, other factors could also influence shell calcification (de Villiers 2004; Beer *et al.* 2010; Naik *et al.* 2013). Oceanic regions dominated by monsoon nurture optimum growth conditions for specific species of planktonic foraminifera during intense monsoon conditions which can be the primary influence on shell calcification and lead to heavier shell weights (Naik *et al.* 2013). There is also a critical gap in understanding the evolution of the carbonate system during the Miocene and Pliocene (Sosdian *et al.* 2018). We therefore attempt to understand the changes in carbonate ion concentration of surface waters by using shell weights of planktonic foraminifera in a region which is presently an atmospheric CO₂ source. We utilize two sites U1467 and U1468 (IODP Expedition 359) covering the last 20 Ma to understand (i) changes in

the carbonate ion concentration of surface waters associated with large climatic shifts of the last 20 Ma, and (ii) the effect of SAM on shell calcification in this region.

2. Study area

The Maldives carbonate platform is situated in a regime of strong seasonal climatic and oceanographic variations related to the Asian monsoonal system. During winter monsoon or the northeast monsoon (November–March), the easterly winds deepen the mixed layer, but the inflow of low salinity water mass from Bay of Bengal (Schulte *et al.* 1999) leads to reduced primary productivity. During summer monsoon or southwest monsoon (June–September) the wind fields have westerly configurations and the current pattern is dominated by the eastward flowing Summer Monsoon Current (SMC) whose velocities increase south of Sri Lanka (Tomczak and Godfrey 2003). A thin low-salinity (5–10 m thick) layer overlies the upwelled water due to local precipitation during summer monsoon periods and hence reduces the effect of upwelling at sea surface though the thermocline is shallow. During the inter-monsoon phase (April–June) an anti-cyclonic circulation is developed in the northwestern Indian Ocean due to strong eastward flow of wind across the equatorial region, whereas the transition to winter monsoon (October–November) witnesses the replacement of East Arabia Current (EAC) by the East Indian Winter jet that flows in opposite direction (eastwards along equator) around southern part of India. Thus the primary productivity reaches its maximum during the inter-monsoon months (Schulte *et al.* 1999) due to stronger Indian Ocean Equatorial Westerlies (IEW).

3. Materials and methods

The samples used in the present study were obtained onboard the JOIDES Resolution, during IODP Expedition 359. The sites U1467 and U1468 were drilled in the Inner Sea of Maldives, equatorial Indian Ocean, at a water depth of 487.4 and 521.45 m, respectively (figure 1a and b). Site U1467 is located at 4°51.02'N, 73°17.02'E, and is the eastern-most site located in the Inner Sea. Site U1468 is located at 4°55.98'N, 073°4.28'E, in the Kardiva channel of Inner Sea. These sites lie within the present day Oxygen Minimum Zone (OMZ)

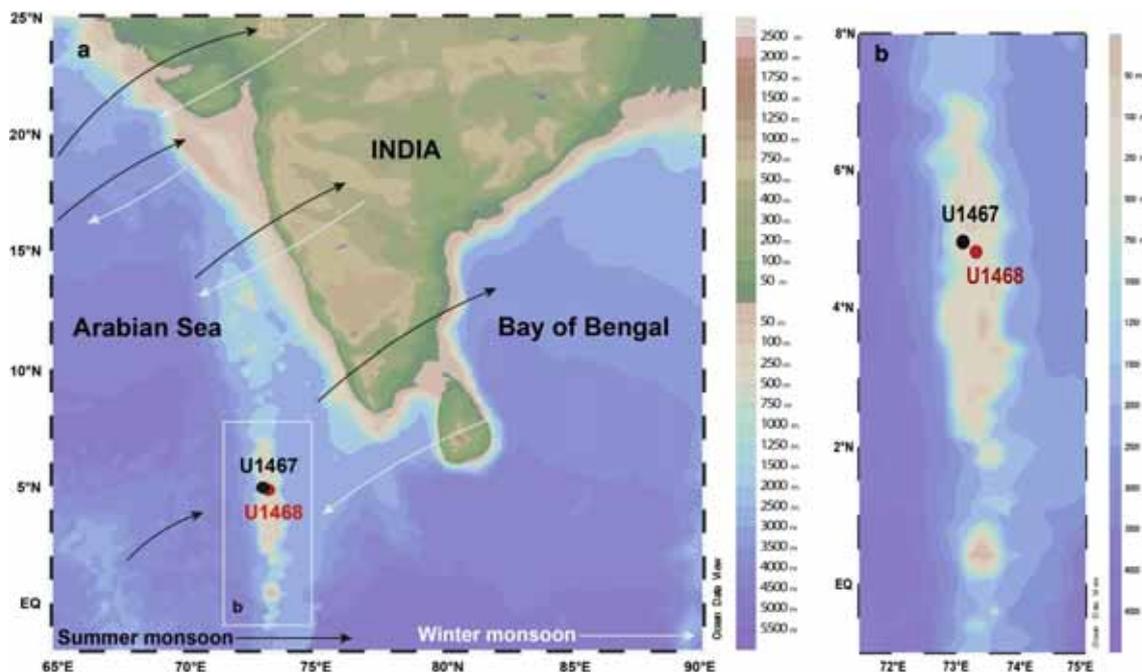


Figure 1. (a) Location of Maldives with major wind directions during the summer and winter monsoon indicated by black and white arrows, respectively. (b) The black and red dots indicate the location of the sites used in this study.

(Betzler *et al.* 2016). These sites were located at 5°S during late Oligocene (~31 Ma) and attained the present position (4.5°–5°N) around 20 Ma (van Hinsbergen *et al.* 2015).

The age model was established by using the biostratigraphic events with respect to the first occurrence (FO) and last occurrence (LO) of well-established diagnostic species which are tied to the geomagnetic polarity timescale of Holbourn *et al.* (2005) (Betzler *et al.* 2016, 2017, 2018). Furthermore, calcareous nannofossils and foraminifera were studied from core sections for a refined age determination (Betzler *et al.* 2016). For the calcareous nannoplankton, the standard zonal scheme of Martini (1971) was adopted as a general frame of reference (Betzler *et al.* 2018). Information on the reliability, definition, and timing of events was provided using the compilations of Backman *et al.* (2012) and Raffi *et al.* (2006). Furthermore, a direct correlation of the seismic stratigraphy and the biostratigraphy was achieved. The core to seismic correlation relies on an accurate P wave velocity model for time–depth conversion (Betzler *et al.* 2018). Dating of the drift onset was a high-priority and this key interval was cored at four sites. An age of 12.9 Ma was arrived at with an uncertainty of ca. ± 0.4 Ma (Betzler *et al.* 2016). In this study, sites U1467 and U1468 were used in conjunction to obtain a total age of 20 Ma.

All analyses were carried out at the CSIR-National Institute of Oceanography, Goa. The sediment samples were disaggregated by soaking in distilled water and were wet sieved with a mesh size of $>63 \mu\text{m}$. The samples were oven dried at about 60°C and the coarser material was further sieved in size range of 300–355 μm . Planktonic foraminifera, *Globigerinoides trilobus* (~50 shells) were picked using a stereo-zoom binocular microscope (Meiji Techno EMZ-5) for shell weight analysis. Care was taken to eliminate shells with overgrowths of secondary calcite or any textural changes due to recrystallization or those with infilling of detrital material and coccoliths. The shells were weighed on a microbalance (Sartorius me5) with a precision of $\pm 1 \mu\text{g}$. The $>125 \mu\text{m}$ fraction was divided by a microsplits to obtain around 300 specimens of foraminifera. The ratio between planktonic (P) and benthonic (B) is expressed as plankton percentage of foraminifera fauna: $[P/(P+B) \times 100\%]$ (Nigam and Henriques 1992).

For the analyses of titanium and aluminum, about 50 mg of sediment was weighed in a Teflon beaker. 10 ml of acid mixture in the ratio (7:3:1) of HF + HNO₃ + HClO₄ was added and heated on a hot plate. Rhodium was used as an internal standard and final solutions were made in an HNO₃ medium. The solution was then diluted to 100 ml by adding ultrapure water (18.2 M Ω). These

solutions were analyzed on a Perkin–Elmer OPTIMA 7300 DV Simultaneous ICP–OES. Marine mud sediment standard (MAG-1) of the United States Geological Survey and stream sediment standard (JSD-1) of Geological Survey of Japan were analyzed along with samples. The precision of measurements as indicated by duplicate samples is $\pm 2\%$ and the accuracy of the analytical results is within $\sim 5\%$. Finely ground sediment samples were used for the analysis of total and inorganic carbon. Inorganic carbon (IC) content was determined with a coulometer (UIC, Inc-CM5130 acidification module) and expressed as $\text{CaCO}_3\%$. Analytical grade calcium carbonate was used as a standard reference material. The precision of measurements were better than $\pm 2\%$. Total carbon (TC) was measured with a Thermo Flash 2000 CHN-elemental analyzer. The results have a precision within $\pm 3\%$ as obtained for NC soil standard. OC% (organic carbon) was calculated as the difference between TC and IC. Scanning electron micrographs (SEMs) were taken on a 6360 LV, JEOL instrument.

4. Results

Globigerinoides trilobus shell weights show maximum values during the colder periods and range from 15.5 to 27.6 μg for the last 20 Ma. Lowest shell weights, of 15.5 μg were noted during the warm MCO and heaviest shells of 27.6 μg at ~ 12.07 Ma (figure 2a). There is also a marked low in shell weights at ~ 10.5 Ma. Heavier shells were observed during the Miocene glaciations (Mi2 to 6) and also during the initiation of the NHG. In general, shell weights show an increasing trend in accordance with decreasing atmospheric CO_2 except for the last 1 Ma where shell weights decrease drastically (figure 2a). Planktonic/benthic ratios were also analysed at key intervals within the core and expressed as planktonic% (figure 2a). The planktonic% was noted to be $>90\%$ in all the samples analysed, which suggests that the site is from a pelagic realm.

Ti/Al ratios range from 0.01 to 0.1 and in general show a gradual increase beginning from ~ 12 Ma (figure 2c). There is a sharp drop in Ti/Al at ~ 5 Ma which recovers instantly and the ratios increase further gradually. The organic carbon content (OC%) varied between 0.2 and 6% with episodic higher values throughout the cores and a major increase in OC% towards 10.5 Ma

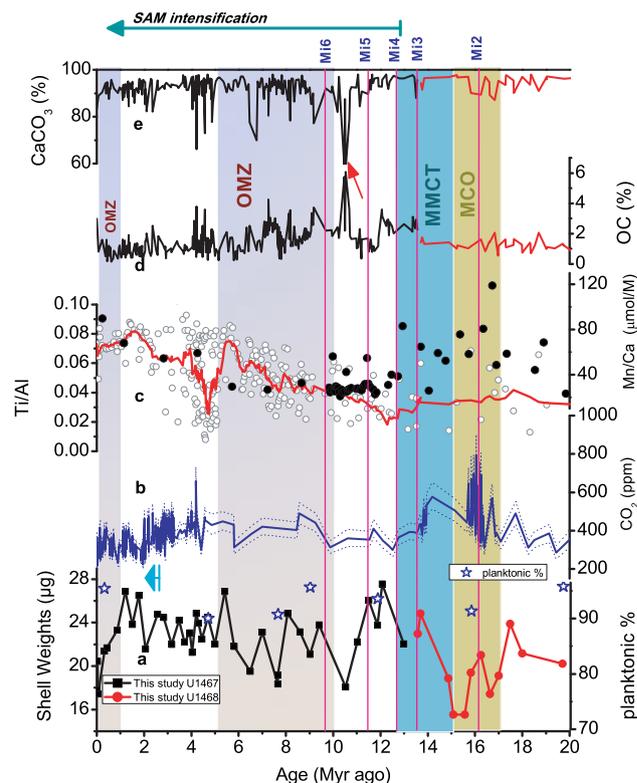


Figure 2. (a) Shell weight of planktic foraminifera *Globigerinoides trilobus* (300–355 μm size fraction) from sites U1467 and U1468 combined to cover the last 20 Ma, and planktonic to benthic ratio expressed as percentage of planktonic foraminifera. (b) Reconstructed CO_2 record from Sosdian *et al.* (2018) compiled to produce an average curve for the last 20 Ma. The dotted lines show 66% confidence intervals. (c) Ti/Al ratios for both the sites, U1467 and U1468 shown as circles. The red line shows running average of the data. Also plotted are the Mn/Ca ratios (Betzler *et al.* 2016) from these sites showing there was a stable OMZ during 10–5 Ma. (d) Organic carbon % for the last 20 Ma. (e) CaCO_3 wt.% for the same period. Red arrow marks the carbonate impoverishment event at ~ 10.5 Ma. The warm Miocene Climatic Optimum (MCO) and cold middle Miocene Climate Transition (MMCT) are marked with yellow and blue bands, respectively. The Miocene glacial events Mi2 to Mi6 (Miller *et al.* 1991) are marked by vertical red lines. South Asian Monsoon (SAM) intensification (Betzler *et al.* 2016) is marked by the green arrow on top of the figure. Period of stable OMZ in the Maldives Sea (Betzler *et al.* 2016) and Arabian Sea (Tripathi *et al.* 2017) are marked by colour gradient bands. The initiation of Northern Hemisphere glaciation (NHG) (Maslin *et al.* 1998) is shown by a blue arrow.

(figure 2d). $\text{CaCO}_3\%$ varied between 58 and 98% (figure 2e). $\text{CaCO}_3\%$ at these sites remained high almost throughout the entire period due to continuous carbonate deposition (Betzler *et al.* 2016), except for some events at ~ 6.5 to 7 and ~ 10 to 10.5 Ma where it dropped to 59% (figure 2e). Both $\text{CaCO}_3\%$ and OC% variations increased after ~ 13 Ma when the sea level controlled sedimentation

changed to a current controlled one due to intensification of the SAM (Betzler *et al.* 2016).

5. Discussion

The Maldives region is largely affected by the monsoon system and its associated wind regime. Shamal winds, northwesterly in direction, develop with the low-pressure monsoon system over India and carry dust from nearby western and north-western deserts up to the equatorial Indian Ocean (Lindhorst *et al.* 2019). The core location is however shielded by atolls from riverine terrestrial input (Betzler *et al.* 2016, 2018; Bunzel *et al.* 2017; Kunkelova *et al.* 2018) and the terrigenous inputs are not so significant (Aubert and Droxler 1992; Purdy and Bertram 1993; Lüdmann *et al.* 2013), especially the fluvial derived material delivered by currents from the Arabian Sea and the Bay of Bengal (Kolla and Rao 1990). We used the Ti/Al elemental ratios to understand terrigenous inputs during the 20 Ma time period. Al and Ti are considered extremely resistant to weathering and are generally used to estimate the abundance of terrigenous materials in sedimentary environments (Murray and Leinen 1996). The Ti/Al ratios from the sites U1467 and U1468 show large variability from 20 to 12 Ma and a gradual increase beginning from ~12 Ma to recent, punctuated by a drop in ratios at ~5 Ma. The increase in Ti/Al beginning at ~12 Ma can be associated with the strengthening of winds and resulting currents associated with initiation of the SAM (Betzler *et al.* 2016). The 10–5 Ma period corresponds to an intense and stable OMZ as seen in the Mn/Ca ratios, which is a result of increased monsoon strength (Betzler *et al.* 2016, figure 2c). This period, based on magnetic susceptibility data shows higher aeolian inputs due to increased wind strength (Betzler *et al.* 2016).

The core site in modern times is a CO₂ source region with an annual sea–air CO₂ flux of 18.7 μatm , highest of which is during the transition (between northeast and southwest monsoon) month of April (Takahashi *et al.* 2009). The planktonic foraminifera *G. trilobus* shell weights were compared to global pCO₂ record which was reconstructed using the following parameters: ‘Fluid inclusions’ seawater composition data, ‘Palike’ CCD reconstruction and G17 boron isotopic composition of seawater (Sosdian *et al.* 2018). The sediment cores used in this reconstruction are mostly from oceanic regions wherein the surface

seawater is in equilibrium with atmospheric CO₂ and hence the reconstructed pCO₂ is a data of atmospheric CO₂ variability (figure 2b). This is the first record of planktonic foraminifera shell weight covering the Miocene to Pleistocene. Broadly, the shell weight record from sites U1467 and Site U1468 follows a trend opposite to the atmospheric CO₂ trend. The MCO was a period of global warmth with increased CO₂ concentrations in the atmosphere of ~600 ppm (Greenop *et al.* 2014) and pH as low as 7.6 (Sosdian *et al.* 2018). The shell weight record during MCO is in general lighter in comparison to the entire 20 Ma record. However, within the MCO there are a couple of shell weight data at ~16 Ma which are slightly heavier. The MCO had large CO₂ variations of up to 400 ppm (Greenop *et al.* 2014) and the slightly heavier shells from the present study correspond to the CO₂ lows within the MCO (figure 2a). The transition from this warmer climate to the modern icehouse world was marked by the MMCT (15–12.5 Ma) where East Antarctic ice sheet (EAIS) expanded and the climate cooled with an associated decline in CO₂ (Foster *et al.* 2012), primarily due to the sequestration of carbon in Monterey Formation of California (Vincent and Berger 1985) and probably some amount due to the silicate weathering of Himalayas (Raymo 1994). This led to an increase in the oceanic pH to ~8.1 at ~12.5 Ma (Sosdian *et al.* 2018). Shell weights show a sharp increase at the beginning of the MMCT and peaking at ~12.06 Ma probably related to increase in surface water [CO₃²⁻] as a result of CO₂ decline. The shell weights in general were heavier during the glacial periods from Mi 2 to Mi 6 (Miller *et al.* 1991) when atmospheric CO₂ was lower.

The period from ~12 to 10.5 Ma shows a steep drop in shell weights with an associated drop in CaCO₃% at ~10.5 Ma. This period between 12 and 10 Ma is documented as an intense carbonate impoverishment period, termed as the ‘carbonate crash’ by Lyle *et al.* (1995), and seen in all major tropical ocean basins with prolonged episodes of reduced carbonate deposition (Lübbers *et al.* 2019). This implies that global changes in the intensity of chemical weathering and riverine input of calcium and carbonate ions into the ocean reservoir were instrumental in driving the carbonate crash (Lübbers *et al.* 2019). An increase in organic export flux is also documented at the same time due to increase in monsoon intensity (Lübbers *et al.* 2019). SEM images at this interval show a clear dissolution of spines in foraminifera (figure 3a).

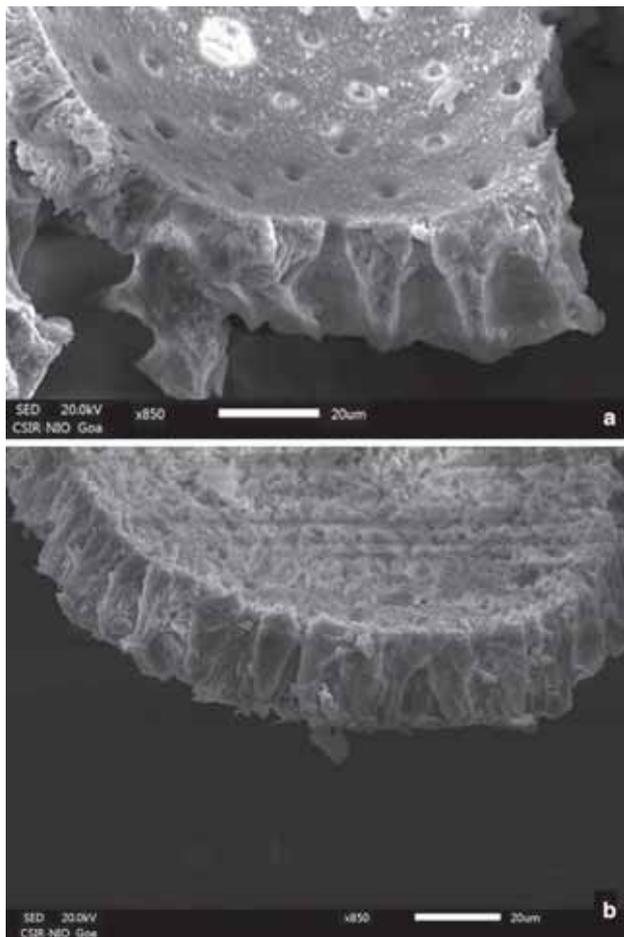


Figure 3. SEM micrographs of *G. trilobus* wall with a magnification of 850 \times at (a) 7.7 Ma, which is from within the intense OMZ period and (b) 10.5 Ma, which is the ‘carbonate crash’ event. Dissolution of spines is seen in both the samples; however, the sample at 10.5 shows relatively more dissolved spines.

Dissolution may take place as the shells pass the sediment–water interface wherein there is an intense organic matter respiration (de Villiers 2005). Betzler *et al.* (2016) have shown the period of ~ 12.9 Ma (which is slightly prior to the 12 Ma period where carbonate dissolution begins) to be the switch from sea-level controlled platform growth to the current-dominated sedimentation. We see a large increase in OC% during the 10 Ma event which is close to the high productivity event of 10–8 Ma observed in the Indian Ocean (Gupta *et al.* 2004).

For the period of ~ 9 to 3 Ma, surface ocean pH remained between ~ 8.0 and 8.1 (Sosdian *et al.* 2018). There is a CO₂ peak at ~ 9 Ma which is not seen in any climate records (Sosdian *et al.* 2018). This peak however reflects in our shell weight records showing a shell weight decrease. This

period also encompasses the CO₂–climate decoupling period as suggested by Pagani *et al.* (1999). Furthermore, the period from ~ 10 to 5 Ma is a period of stable OMZ conditions as seen from Mn/Ca elemental ratios at sites U1466, U1468 and U1471 (Betzler *et al.* 2016, figure 2c). Mn/Ca ratios in general show a drop from 20 Ma up to ~ 10 Ma (Betzler *et al.* 2016). After which they remain low up to 5 Ma, indicating that this was the time of intense OMZ in the Maldives Sea. It has been shown that OMZs are associated with high CO₂ which is defined as a carbon maximum zone (CMZ) (Paulmier *et al.* 2011). The present cores being recovered from the OMZ will have a buildup of carbon during this period of intense and stable OMZ. Superimposed on the anti-correlation with CO₂ at specific periods, shell weights during this period from 10 to 5 Ma show large fluctuations and are on an average lighter, probably as a result of calcite dissolution after settling into sediments as a result of build-up of CO₂ in bottom waters. SEM micrograph of *G. trilobus* shell wall at 7.7 Ma shows dissolution of spines but with relatively less intensity in comparison to shells at 10.5 Ma (figure 3b). Furthermore, the OMZ is known to reach its modern strength at about ~ 1.0 Ma closely following the enhanced surface water productivity (Tripathi *et al.* 2017) which implies that SAM intensified from ~ 1.0 Ma (Derry and France-Lanord 1996; Clift *et al.* 2008). The increased OMZ intensity from ~ 1.0 Ma again created conditions favourable for increase in bottom water CO₂ leading to dissolution of shells which reflects in lower shell weights for the past 1 Ma.

Early Pliocene $\delta^{11}\text{B}$ based pCO₂ values between 4.6 and 3.6 Ma were above a suggested threshold of 280 ppm below which Northern Hemisphere glaciations are possible (De Conto *et al.* 2008). This was a period of prolonged warmth terminating at ~ 2.7 Ma. Atmospheric CO₂ decreased to minimum of 245 ppm at 3.32 Ma after which the Northern Hemisphere glaciations began (Bartoli *et al.* 2011) and subsequently lead to the glacial–interglacial climate variability in the Pleistocene (Lisiecki and Raymo 2005). Shell weights from our core sites remained low from ~ 5 to 3 Ma after which they increased in conjunction with the decrease in atmospheric CO₂ and the initiation of NHG, peaking at ~ 1 Ma and a sharp decrease thereafter.

Finally, in order to look at a natural analogue of the near future of ocean acidification, we observed shell weights during the MCO, the period of highest atmospheric CO₂ concentrations during the last

20 Ma. Lightest shell weights of $\sim 15.5 \mu\text{g}$ occurred at 15.08 Ma when atmospheric CO_2 was ~ 500 ppm (figure 2a and b) during the MCO. In comparison, pre-industrial shell weights were $\sim 17 \mu\text{g}$ with atmospheric CO_2 concentrations at ~ 280 ppm. However, we do not have any record of present day shell weights in this region which would be an interesting comparison to elucidate ocean acidification.

6. Conclusions

The Ti/Al ratios from sites U1467 and U1468, suggest an increase in terrigenous inputs to the Maldives Sea beginning from ~ 12 Ma which is in line with strengthening of winds associated with initiation of SAM. Shell weights of *G. trilobus* from these sites negatively co-vary with global atmospheric CO_2 throughout most of the period from Miocene to Pleistocene which suggests that the Maldives Sea behaved similar to other tropical regions of the world throughout the 20 Ma period in terms of its carbonate chemistry. It also shows that the primary control on shell calcification is surface water carbonate ion concentration. The carbonate impoverishment as seen in all major tropical ocean basins between 12 and 10 Ma is documented in the present study by lighter foraminifera shells and visual dissolution of spines. During periods of intense OMZ, shell dissolution due to corrosive bottom waters appears to obscure the understanding of surface water carbonate ion concentrations.

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