

EUG XI



Symposium CC07

Biomarkers in Palaeoceanography and Palaeolimnology

Convenors

Joan Grimalt
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Monday PM Session

CC07 : MOPm21 : G2

Long-Chain Alkenones in Limnic Systems, Paleothermometers or Not?

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The distribution of long-chain alkenones (LCA) in marine sediments allows to reconstruct paleo-sea-surface temperatures (Brassell et al., 1986; Prahil & Wakeham, 1987). Our study investigates the potential of this approach in lacustrine systems linking the marine and the terrestrial climatic record. Previously Cranwell (1985), Thiel et al. (1997), and others reported LCA in limnic systems without relationship between LCA distribution and lake temperatures. Source organisms could not be identified but a preference of tetra-unsaturated alkenones in limnic systems was observed. Sediments from 13 out of 30 lakes (Germany, Austria, U.S., Russia) yielded high abundances of C₃₇- and C₃₈-LCA with known predominance of tetra-unsaturated compounds. LCA patterns of Recent lake sediments and averaged lake summer surface water temperatures covariate (r^2 for Uk'37 = 0.90, r^2 for Uk'37 = 0.67). Hence, the same mechanism that causes temperature-dependence of LCA patterns in marine systems might be effective in limnic settings. An empirical relationship between LCA distribution pattern and temperature was observed for fossil (Younger Dryas) sediments from Lake Steisslingen, SW-Germany. A good correspondence between Uk37 and $\delta^{18}O$ -isotope of lake chalk occurs. In surface water samples from the "Brandenburg-Mecklenburger Seenplatte" LCA were not detected. Water samples taken at the chlorophyll maximum in about 14 to 17 m water depth of Lake Stechlin, NE-Germany, yielded LCAs in medium abundances. LCA for the first time were identified in the water column of a fresh water lake, indicating a biosynthetic origin from phototrophic (micro)algae. Cultivation and lipid analyses of potential freshwater LCA-producing algae failed but we can exclude several species and strains as LCA-precursor by analyses or their abundance in lakes without LCA. All fresh water haptophytes were tested negatively. A reason for the difficulties to detect the alkenone producers might be their small size and sensibility against preparation methods.

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CC07 : MOPm22 : G2

Hopanooids as Environmental Markers

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Hopanooids are important biomarkers, in that they occur virtually wherever there is organic matter in the geosphere (Ourisson and Albrecht, 1992). They are derived from bacteriohopanepolyols that are synthesised as membrane lipids by a diverse range of bacteria. Different bacterial groups can possess recognisable bacteriohopanepolyol distributions (Rohmer et al., 1984), affording these compounds potential as markers for specific bacterial populations and thus environmental conditions.

Here we report the findings of a study that determined the abundance and composition of the bacterially-derived bacteriohopanepolyols in Recent sediments from a large suite of depositional environments. These compounds are routinely determined by GC-MS following cleavage of the polyfunctionalised side chain with periodic acid/sodium borohydride (to form simple hopanol products that are characteristic of different groups of bacteriohopanepolyols). Our data show that tetrafunctionalised compounds, including bacteriohopanetetrol and hopanooids with a more complex terminal functional group, dominate the bacteriohopanepolyols of marine sediments. In contrast, lacustrine sediments are characterised by high relative abundances of bacteriohopanepolyols with five and six

functional groups on the side chain, characteristic of methanotrophic bacteria (Farrimond et al., 2000). This feature is especially marked in the sediments of small eutrophic lakes with high methane fluxes.

We have recently developed an HPLC-based method for the analysis of intact bacteriohopanepolyols (i.e. without side-chain cleavage). Analysis of a suite of methanotrophic bacteria confirm that their hopanooids are dominated by penta- or hexafunctionalised hopanooids, and that the two types of methanotrophs recognised by microbiologists can be distinguished by their hopanol composition: Type I methanotrophs (abundant in aquatic environments with high methane fluxes) produce almost exclusively hexafunctionalised biohopanooids, whilst Type II methanotrophs (characteristic of terrestrial environments such as soils and peats) synthesise penta- (mainly) and tetrafunctionalised biohopanooids. Application of the HPLC method to sediments is allowing us to characterise depositional environments in terms of the dominant bacterial inputs of hopanooids, these being: (1) water column Type I methanotrophs (small methane-rich water bodies), (2) terrestrial Type II methanotrophs (soil/peat input to aquatic systems), and (3) other, less specific bacterial inputs (which dominate marine settings).

The pentacyclic nucleus of the hopanooids is relatively stable and may survive diagenesis in a recognisable form, but often the more diagnostic functionalised side chain is lost due to cleavage reactions. A variety of geohopanol products are identifiable in sediments including acids, hopanols, hopenes and ultimately the hopane biomarkers that are routinely employed in petroleum geochemistry and organic geochemical studies of ancient sedimentary rocks. The ubiquity of these geohopanooids in sediments and the source specificity of some of the precursors and their early diagenetic products suggest that they have the potential to preserve valuable palaeoenvironmental information in the sedimentary record.

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CC07 : MOPm23 : G2

Molecular Markers of Land Use Change

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Biological markers, particularly lipids, have often been employed to provide historical information of organic matter (OM) accumulation in lacustrine sediments (eg. Meyers et al., 1984; Ho and Meyers, 1994). More subtle changes in OM inputs, for example from changes in catchment vegetation, have been less frequently observed, but potentially, differences in distributions of certain compound classes can be informative (e.g. n-alkanes; Cranwell et al., 1987; van Bergen et al., 1997). Here, we will show that the organic geochemistry of the sediments combined with classical palaeolimnological data leads to convincing evidence of a changing environment in the catchment area of a small lake reflects over the past 3000 years. Gormire lake was chosen as the study site; this is a small landscape lake on the western edge of the Humberdon Hills (Yorkshire, U.K.), which has a steep, well-defined catchment, underlain by Lower Jurassic rocks and has undergone periods of land use change and deforestation during the Holocene period. Simple biomarker parameters, such as the mean carbon number of n-alkanes and the ratio of lignin thermochemolysis products are employed, together with classical pollen analysis and bulk organic geochemistry, to assess qualitative and semi-quantitative changes in OM inputs to the lake sediments.

1. Comparison with the pollen inputs shows that during periods of deforestation, which commenced some 2735 and 1480 BP, shows there was a significant decrease in organic carbon contents of the lake sediments, which reflects dilution by enhanced influx of clastic material. 2. The mean carbon number of n-alkanes, which are dominated by high molecular weight (>C₂₅) homologues vs sediment depth

shows an extremely close correspondence with the percentage of pollen derived from trees and shrubs in the sediments. This reflects the more waxy nature of epicuticular waxes of grasses when compared to trees. A higher-plant derived triterpenoid, tentatively identified as 28-carboxyursen-12-enol, appears exclusively in samples where tree and shrub-derived pollen is dominant. It appears to be a specific marker for OM derived from these plants. 3. Thermochemolysis of lignin, which allows source specific analysis of the bulk component of the allochthonous organic material in the sediments, confirms that there was a significant change in the nature of material deposited in the lake sediments from woody to grass dominated, which is contemporaneous with the deforestation event. 4. There is evidence from both the sedimentary C/N ratios and yields of cinnamyl, vanillyl and syringyl phenols derived from lignin thermochemolysis, to suggest that the deforestation events have led to enhanced aquatic productivity, beginning with the development of reed-swamp vegetation. This may reflect an enhanced influx of soil associated nutrient to the lake. In more recent years, there is also some evidence of increasing phytoplankton productivity in the lake.

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CC07 : MOPm24 : G2

Lipid Biomarkers as Indicators of Local Environmental Conditions in a Terrestrial Ecosystem during the Late Pleistocene and Holocene: A Case Study on Maar Lakes of the Eifel-Volcanic-Field, Germany

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Maar lakes are formed upon subterranean volcanic eruptions and in most cases are characterised by small catchment areas. As a result of the small ratio of diameter vs. depth, oxygen supply to bottom waters is often limited leading to an anoxic hypolimnion and enhanced organic matter (OM) preservation. Therefore, maar lakes may be regarded as ideal archives for paleolimnological studies using organic-geochemical methods. Here we report on lipid distributions in Lake Holzmaar and Lake Meerfelder Maar in the Eifel-Volcanic-Field, Germany. The two maar lakes are only 10 km apart, giving an excellent opportunity to investigate the influence of local variations on the sedimentary OM composition.

Samples from a 10 m sediment core from Lake Holzmaar and a 11 m sediment core from Lake Meerfelder Maar have been included in this study, representing the last 12,500 and 15,000 years, respectively. Dating of sediments is based on varve counting and ¹⁴C-age determinations. Isolated lipid class fractions were analysed by gas chromatography and gas chromatography-mass spectrometry.

The lipid biomarkers were evaluated with respect to their significance as indicators of organic matter sources. A strong shift of the $n-C_{27}/(n-C_{27}+n-C_{29})$ alkane ratio in the lowermost samples from Lake Meerfelder Maar indicates a vegetation type change from herbaceous to forest vegetation in agreement with pollen data. The medium-chain n-alkanes originating from macrophytes systematically increase between 15,000 and 10,000 years BP and then remain at a relatively high level until 4,000 years BP after which another decrease is observed. As expected, the variation for the long-chain land plant n-alkanes follows the opposite trend. In Lake Holzmaar the relative amounts of macrophyte n-alkanes is almost generally much lower than in Lake Meerfelder Maar showing a decreasing trend to present age. For both lakes, variations complementary to those of the n-alkanes are observed for the fatty acid distributions. In general, short-chain fatty acids (C₁₆, C₁₈) are of low abundance, probably underestimating the algal contribution to the sedimentary organic matter. The role of these

primary producers will be further evaluated using more suitable lipid biomarkers such as the sterols. The sources of organic matter to both maar lakes are strongly variable over time and, moreover, significant differences exist between the two lakes. Macrophytes seem to have played an important role in Lake Meerfelder Maar in particular during the Holocene optimum whereas they obviously never established as an important source of OM in Lake Holzmaar. This and the higher relative algal contribution as revealed by other biomarker proxies might indicate a higher trophic state of Lake Meerfelder Maar *versus* Lake Holzmaar during the Holocene.

CC07 : MOPm25 : G2 A Biomarker Record of Holocene Environmental Changes in the Mediterranean Region- Lake Albano (Central Italy)

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Understanding the causes of Holocene environmental changes depends on the development of reliable proxies that can be applied to securely-dated sedimentary sequences. Organic matter (OM) compositional variations in the predominantly laminated, organic carbon-rich sequence of Lake Albano illustrate the response of an aquatic system to these environmental changes. Thus, the results of our biomarkers study combined with other OM bulk parameters provide new insights into the possible forcing mechanisms of Holocene environmental variations.

Three distinctive intervals can be identified within the Holocene: (i) 0-3.8 ka BP, (ii) 3.8-6.9 ka BP and (iii) 6.9-11.48 ka BP. Zone (i) displays high values of dinosterol, tetrahymanol, lanosterol and n-alkanes. It also shows high contents of certain pigments such as echinenone and isorenieratene as well as high hydrogen index (HI), whereas diadinoxanthin concentrations decrease. Conversely, zone (ii) shows very low concentrations of dinosterol, tetrahymanol and lanosterol. Decreasing contents of n-alkanes correlate with low amounts of echinenone, isorenieratene and low HI, whereas diadinoxanthin and Oxygen Index (OI) reach maximum values. Zone (iii) is similar to zone (i) with respect to pigment and dinosterol concentrations, but exhibits lower values of tetrahymanol, lanosterol and n-alkanes.

Diadinoxanthin and OI indicate a dominance of diatoms as primary producers, whereas dinosterol and echinenone suggest dinoflagellates and cyanobacteria, respectively. Dinoflagellates prefer stable water column stratification (zones i and iii) whereas diatoms are adapted to more turbulent conditions (zone ii). Water column stability in zone (iii) is further indicated by high isorenieratene contents, a diagnostic pigment for phototrophic bacteria growing under strictly anoxic conditions in the hypolimnion. Moreover, increasing concentrations of tetrahymanol, an organic compound produced by ciliates, can be attributed to the presence of a well-developed oxic-anoxic boundary. The latter indicates more stable anoxic conditions during zone (i) compared to the diatom rich zone (ii).

Variations on wind intensity, precipitation and temperature are the dominant factors affecting OM production in aquatic environments. Changes in these parameters between 6 and 7 ka BP are commonly observed at the end of the early Holocene climate optimum. At ca. 4.0 ka BP a further climatic deterioration is indicated by neoglaciations in the Alps. During this time interval, however, distinctive alterations in aquatic systems have been also associated with deforestation and other human-induced environmental disturbances in densely populated areas of the world such as the Lazio region. This is consistent with the observed increasing lanosterol and n-alkanes concentrations in Lake Albano OM after ca. 4 ka BP, which are both indicative of increased input of higher land plants and thus soil erosion.

CC07 : MOPm26 : G2 Origin, Transport and Surface Distribution of Terrigenous Organic Matter in the Equatorial Atlantic: Evidence from Lignin Chemistry, Carbon Isotopes, Pyrolysis and Petrology

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The importance and fate of terrigenous organic matter (OM) in the ocean still remains one of the central questions in the global carbon cycle since only a small fraction of the OM preserved in deep-sea sediments appears to be land-derived. However, recent results from the Amazon deep-sea fan and from surface sediments of the pelagic low latitude Atlantic off West-Africa recommend a general re-evaluation of the popular assumption that deposition of terrigenous organic carbon is restricted to deltas and continental margins, and rarely occurs beyond the shelf-slope boundary. This critical point especially holds true for late Quaternary glacial periods when sea-level was lowered and continental dust supply was enhanced. For the discussion of terrestrial OM in marine environments, the Equatorial Atlantic serves as an interesting study area. Its model character is related to the pronounced asymmetry between the western, central, and eastern low latitude Atlantic with regard to source areas, transport mechanisms and distances, and flux rates of terrigenous organic carbon. We applied a multiparameter approach combining terrestrial biomarker, carbon isotopes, Rock-Eval Pyrolysis and organic petrology on surface sediments from the African continental margins between Congo and Nigeria as well as the central deep Equatorial Atlantic. Depositional settings studied cover the river-dominated Congo and Niger deep-sea fans with their corresponding continental margins and the dust-influenced sector of the equatorial oceanic upwelling system. Supply of terrigenous OM is highest along the river-influenced upper continental margins along equatorial Africa (approaching up to 80% of the bulk OM off Gabon) but still comprises a considerable share in remote areas of the central Equatorial Atlantic (about 20% of bulk OM). Terrestrial biomarker, pyrolysis and petrological data consistently indicate a change in the composition of terrigenous OM from shallow to deep-marine settings. Accordingly, river-influenced continental margins are typically characterized by a mixture of mainly non-oxidized terrigenous OM of angiosperm origin (high S/V, low C/V lignin-phenol ratios) whereas the dust-influenced central Equatorial Atlantic reveals a more inert (stronger oxidized) geochemical signature. Lignin-phenols in pelagic sediments indicate an enhanced admixture of gymnosperm higher plant tissue including some C4 grass debris (high C/V, high S/V lignin-phenol ratios). These general differences in concentration and composition of terrigenous OM obviously relate to contrasting transport mechanism (eolian vs. fluvial), transport distances and vegetative cover of the corresponding continental source areas.

CC07 : MOPm29 : G2 Late Quaternary Variations of Terrigenous Organic Matter Composition on the Congo Deep-Sea Fan (ODP-site 1075): An Organic Geochemical Multiparameter Approach

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Terrigenous organic matter in deep-sea settings still remains an important target of discussion, especially regarding its role within the global organic carbon cycle. The presented multiparameter approach performed on late

Quaternary sediments of the Congo fan illustrates the complexity of climate controlled fluctuations in terrigenous organic carbon supply (C₃ vs. C₄ plant material, low-mature vs. high-mature organic matter), primary productivity and burial of organic carbon in the deep sea.

To characterize the organic matter (OM) composition with regard to terrigenous and marine fractions we used several organic geochemical methods: elemental analysis, lignin chemistry, stable carbon isotopes, Rock-Eval pyrolysis, and organic petrology.

The ratios of specific lignin phenols (S/V, C/V; S = sinapyl, C = cinnamyl, V = vanillyl phenols) demonstrate that the terrigenous OM on the Congo fan is derived from a mixture of woody and non-woody angiosperm tissues, mainly leaves and bark. Within the Quaternary record of ODP-site 1075, varying S/V and C/V ratios reveal the changing composition of the sedimentary terrigenous OM in response to the vegetation cover of the catchment area and the transport mechanism (fluvial, aeolian). Results from organic petrology indicate significant higher shares of terrigenous OM than estimated from the carbon isotopic signature of the bulk organic matter ($\delta^{13}C_{org}$). Application of a binary mixing model to the isotopic data leads to the conclusion that the terrigenous organic fraction comprises 30 ± 5% of the bulk OM which appears to be quite low considering the estimates from petrology (60 to 70%), the presence of mixed kerogen type III/II and the position of site 1075. One reason for an underestimation of the terrigenous proportion based on the isotopic signature may be the contribution of C4 plant material (e.g. tropical grasses) which is isotopically heavier (-12‰) than marine OM (-18 to -20‰).

CC07 : MOPm30 : G2 Tracking Paleohydrologic Change in the Mediterranean Sea using Compound Specific Hydrogen Isotopes

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Hypotheses explaining the origin of deep-sea sapropels agree that deposition of these sediments coincides with intervals of increased seasonality and that their occurrence is related to freshening of the Mediterranean surface waters. In a previous study, we were able to quantify the change in the oxygen isotopic composition of Mediterranean seawater at Sapropel Event 1 (S1) to be 0.8‰, suggesting a decrease in salinity of 1.95-2.96 psu. To constrain the magnitude of the depletion in δD that occurred in conjunction with the excursion in $\delta^{18}O$, and thus quantify the total paleohydrologic change that occurred in the Mediterranean region, we have examined the hydrogen isotopic composition of individual lipid compounds extracted from an eastern Mediterranean Sea sediment core. We focus on the analysis of the D/H ratios in the lipid fraction of the organic matter (specifically n-alkanes and alkenones) as bulk lipids have been shown to accurately record the D/H ratio of surrounding environmental waters in fresh-water aquatic plants (Sternberg, 1988).

To determine the δD of seawater from the measurement of long-chain n-alkanes and alkenones, we use the previously derived offset of 158‰ (Estep and Hoering, 1980; Sessions *et al.*, 1999) between bulk n-alkanes and source water and the recently determined offset of 260‰ between C_{37:2} alkenones and the water in which they grew. To test the method, we first measured the δD of organic compounds extracted from a surface sediment sample and compared it to the modern hydrogen isotopic composition of surface seawater above the core sampling location. Results of the n-alkane analyses show very good agreement (average δD of 10.67‰ compared to average surface water δD of 9.10‰) and additionally indicate that the n-alkanes being analyzed are actually marine in origin and not terrestrially derived (despite the strong odd over even dominance and the predominance of higher carbon number molecules). Reconstruction of the δD of seawater for S1 suggests a depletion of approximately 30-35‰ compared to the present. Results from the n-alkanes (average δD of -21.38‰) and the alkenones (-20.98‰) provide very similar estimates, again signifying that the n-alkanes have a predominantly marine source. These analyses demonstrate the huge potential for applying compound-specific hydrogen isotopes to paleoclimatic reconstructions.

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CC07 : MOp31 : G2

The Carbon Associated Flux of Alkenones in the North Atlantic

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The determination and understanding of the fluxes of natural and anthropogenic chemicals into the deep ocean and their relation to surface biological and hydrographic processes are major goals of many biogeochemical programmes. We have studied the particulate fluxes of biogenic (amino acids, alkenones) and anthropogenic (e.g. Polychlorinated biphenyls) trace organics in the northeast Atlantic. Time-series sediment traps were moored in different depths at 33°N, 47°N and 54°N along 20°W and at 70°N, 4°E between March 1992 and September 1997. Our results confirm the distinction of three biogeochemical provinces with a border line at approximate 40°N: the subtropical gyre and the North Atlantic drift region and at 60°N: the Polar Atlantic region. We observed that the flux of trace chemical substances follows the seasonal cycle of biological production in surface waters, with a shifted flux maximum of about 2-3 month in these three provinces. The transport of alkenones into the deep ocean is associated with the particle flux of organic carbon and calcium carbonate. However, for a number of chemical substances no single relationship with POC could be evaluated throughout the water column. The alkenone flux varied between 30 ng/m²d and 9000 ng/m²d. Strong seasonality were observed at all stations. Implications of the various alkenone fluxes on the alkenone signals in the sedimentary record will be discussed and a new Uk37-index calibration from sediment trap data will be presented.

CC07 : MOp32 : G2

Long and Short Term Variability of Glacial Latitudinal Temperature Gradients and Terrestrial Inputs in the North Atlantic as Recorded in Sedimentary C37 Alkenones and N-Alkanes

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Sea surface temperatures and input of continental materials have been reconstructed from the study of the long chain alkenones and n-alkanes, respectively, in two cores located in the western side of the Mid-Atlantic Ridge (MD952037, 37°05'N 32°02'W, 2630 m depth and SU90/08, 43°30'N, 30°24'W, 3100 m depth). Both the long and short-term variability recorded by the temperatures and the planktonic $\delta^{18}\text{O}$ are basically the same over the last 280 kyr, showing a clear glacial/interglacial evolution. The SST parallelism at both sites is not maintained at IS2 and IS3, when there is evidence for the development of a steep north-south SST gradient. This structure was not existent during IS6, indicating different climatic conditions during these glacial periods.

Comparison with core SU90/08, located only 6° north and directly influenced by the cold polar waters associated to the polar front during glacial times, reveal different climatic conditions during glacial periods at both locations. Whereas core MD952037 record similar SST values during the last two glacial periods (ca 14-15°C), the northern core display colder conditions during isotopic stage 2 (8-10°C) than stage 6 (13-15°C). These results indicate the existence of a well-developed steep north-south gradient between 37 and 43°N during the last glacial period but not during stage 6, which suggest a southern expansion of the polar front during the last glacial maximum. Moreover, a closer inspection of the SST record during the last two glacial periods also suggests a decrease in the northward transport of warm tropical waters, allowing the accumulation of heat in the subtropical ocean at sites located just south of the polar front.

Examination of the long chain odd n-alkanes and the non-carbonate fraction show higher abundances of terrestrial material in core SU90/08 during the last glacial period in concordance with the lowest SSTs observed. In contrast, core MD952037 only present slightly higher abundances during the glacial stages compared to the interglacials, most likely related to eolian sources.

CC07 : MOp33 : G2

Alkenone Measurements of Sea Surface Temperature in the Southeastern Subtropical Pacific Ocean over the Last 400 kyr

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The present study describes the first sea surface temperature (SST) reconstruction in the southeastern subtropical Pacific Ocean, offshore the South American coast. The oceanic circulation in this area is mainly controlled by the Peru-Chile Current system as a part of the South Pacific Subtropical gyre. At about 20°S latitude, the Chile Current partially feeds the Peru Current and turns northward where it is incorporated into the South Equatorial Current. Changes in this circulation system may be dependent on insolation southern hemisphere variability.

Unfortunately, the extremely low sedimentation rates and poor carbonate preservation in this area (most of the southeastern Pacific plates are deeper than 4000 meters, the approximate the present carbonate compensation depth) have prevented the elaboration of SST paleoceanographic reconstructions. Probably, the sediments deposited on the ridges (e.g. Nazca Ridge) offer one of the few chances for overcoming these calcium carbonate dissolution problems.

The record described in the present study encompasses the last 400 kyr and follows the characteristic glacial/interglacial pattern defined by global ice volume but leads the $\delta^{18}\text{O}$ isotopic record by about 10-20 kyr depending on the climatic period. SST in the Holocene is lower by about 0.5-0.8°C than maximal SST in stages 5e, 7, 9 and 11. SST in stages 2-4 is lower by about 0.55-1.3°C than minimal SST in stages 6, 8 and 10. These features are similar to those SST records obtained in the south Atlantic Ocean pointing to a general inter-basin behaviour at these low latitudes. For most of the record, the observed long-term SST evolution is well correlated with the orbital parameter of eccentricity, which modulates the insolation at low latitudes. However, at low eccentricity values (Stages 11 - 9 and Termination I), SST is driven by obliquity, exhibiting a dependence from high latitude climatic responses.