



Sedimentary pigments and nature of organic matter within the oxygen minimum zone (OMZ) of the Eastern Arabian Sea (Indian margin)

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ABSTRACT

Sedimentary pigments, carbon and nitrogen content and their stable isotopes were studied in three short cores collected from the oxygen minimum zone (OMZ) of the Eastern Arabian Sea (EAS). Nine pigments including chlorophyll *a* and their degradation products were quantified using High Performance Liquid Chromatography (HPLC). Astaxanthin followed by canthaxanthin and zeaxanthin were the major carotenoids detected in these cores. The total pigment concentration was high in the core collected from 500 m water depth ($6.5 \mu\text{gg}^{-1}$) followed by 800 m ($1.7 \mu\text{gg}^{-1}$) and 1100 m ($1.1 \mu\text{gg}^{-1}$) depths respectively. The organic carbon did not have considerable control on sedimentary pigments preservation. Pigment degradation was comparatively high in the core collected from the 800 m site which depended not only the bottom dissolved oxygen levels, but also on the faunal activity. As reported earlier, the bottom water dissolved oxygen and presence of fauna have good control on the organic carbon accumulation and preservation at Indian margin OMZ sediments. The C/N ratios and $\delta^{13}\text{C}$ values for all the cores conclude the marine origin of organic matter and $\delta^{15}\text{N}$ profiles revealed signature of upwelling associated denitrification within the water column.

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1. Introduction

Oxygen Minimum Zones (OMZ) are important in terms of organic matter (OM) accumulation, preservation (Schulte et al., 2000) and burial as well as benthic solute fluxes (Berner, 1982; Walsh, 1991; Hedges and Keil, 1995; Woulds and Cowie, 2009). Assessment of organic carbon (C_{org}) and nitrogen fluxes in marine sediments are essential for balancing the global carbon budget and quantification of C_{org} burial and nitrogen removal mechanisms. Arabian Sea OMZ sediment is found to be associated with OM rich deposits in the geological records. It is associated with different groups of benthic communities and degrees of bioturbation in terms of OM preservation majorly due to difference in oxygen availability (Cowie, 2005; Cowie et al., 2009; Hunter et al., 2011, 2012). Sedimentary OM derived from various plankton species is

the major reservoir of C_{org} in the global carbon cycle. Only 1% of the originally produced OM is estimated to be transferred to the deep biosphere (Middelburg and Meysman, 2007).

Sedimentary pigments can provide historical information on the overall primary production in the overlying surface waters as well as the dominant classes of phytoplankton. It was used as paleo-tracers for algal and bacterial communities (Sanger, 1988; Leavitt and Brown, 1988; Leavitt, 1993; Bianchi et al., 2000; Leavitt and Hodgson, 2001) and as an indicator for the state of decay of OM in sediments (Woulds and Cowie, 2009). Moreover, sedimentary pigment biomarkers reflect long term bloom record in estuarine systems with anoxic, laminated sediments (Bianchi et al., 2002). The comparative lability and source specificity makes them a powerful tool used to investigate many aspects of benthic biogeochemical processes as well as OM source and history (Jeffrey and Vesik, 1997; Boon and Duineveld, 1998). The chlorophyll *a* profiles could be useful for an estimation of total flux of OM and it can provide an insight in to the knowledge of degree of bioturbation in the sediments (Boon and Duineveld, 1998). Specific chlorophyll degradation products, such as the pheopigments and esters have been used as markers for grazing activity of zooplankton through

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the water column (Chen et al., 2003). The pheopigment/Chl *a* ratio was used as an index of Chl *a* decay (Bianchi et al., 2002) and Chl *a*/Pheophytin *a* ratio could be an indicator of preservation conditions (Reuss et al., 2010). In general, carotenoids in sediments can be diagnostic of phytoplankton OM source (Millie et al., 1993; Jeffrey and Vesk, 1997; Leavitt and Hodgson, 2001). The redox condition (anoxic) can enhance the preservation of labile pigments in sediments and could be useful for their historical reconstruction (Repeta and Gagosian, 1987; Bianchi et al., 2000).

North Eastern Arabian Sea (NEAS) is characterized by an extremely stable OMZ between 200 and 1200 m water depths with dissolved oxygen values reach up to 0–0.5 ml/l and a strong, seasonal and monsoon controlled variability of primary productivity (Schulte et al., 1999; Naqvi et al., 2003). An Intense OMZ in the NEAS is associated with laminated and less bioturbated sediments that are ideally suited for the paleoceanographic reconstruction with fine resolution. It showed strong relationship with oxygen concentration and distribution of organisms, though biological responses to the OMZ varied with the type of organisms (US JGOFS study). Sedimentary pigment concentration in the Arabian Sea were positively correlated with both abundance and biomass of metazoan macrofauna within the OMZ and negatively correlated with species richness and evenness below the OMZ (Cowie, 2005). Benthic communities within the Arabian Sea OMZ mainly composed of surface-feeding polychaetes and crustacea (Levin et al., 2000). In addition, OMZ in the Arabian Sea is one of the three major denitrification sites in the world Ocean with an estimated annual rate of 10–30 Tg N y⁻¹ (Naqvi et al., 1992). Its contribution to total oceanic pelagic denitrification (80–150 Tg N y⁻¹) is substantial and globally significant (Codispoti et al., 2001). Sedimentary denitrification over the Indian continental shelf has yielded rates of 0.4–3.5 Tg N y⁻¹, which are of same order as reported from other areas (Naik, 2002). Geological records reveal the human-induced perturbations of oxygen and carbon budgets which had potential impacts on the biological diversity and chemical fluxes of the Arabian Sea OMZ (Altabet et al., 2002). High primary production and sediment accumulation rates on the Arabian Sea's margin produce exceptional records of past changes in climatic and oceanographic conditions (Ivanochko, 2004).

Two previous reports on sedimentary pigments from the Arabian Sea were from Oman (Shankle et al., 2002) and Pakistan margin (Woulds and Cowie, 2009) OMZs whereas studies from the Indian margin are not yet been reported. Water depth and bottom DO controlled the chlorin concentration in Oman margin sediments with highly degraded pigment suites, whereas bottom DO and faunal influence played a crucial role in preservation of pigments (less degraded) at Pakistan margin OMZ. We were looking for any similarity or behavioral changes of phytoplankton pigments assemblage in Indian margin compared to other OMZ margins of the Arabian Sea. We have analyzed three short sediment cores from varying water depths (500, 800 and 1100 m) within the OMZ of the north eastern Arabian Sea (NEAS). The objectives of the study include 1) the comparison of phytoplankton pigments assemblage and their degree of degradation in three different sediment cores from varying bottom water oxygen concentrations and 2) the study of the source and preservation of organic matter using the bulk productivity signals C_{org} and CaCO₃ variation within the OMZ.

2. Materials and methods

2.1. Sampling

Short sediment cores were collected from three water depths within the oxygen minimum zone (OMZ) of the NEAS during the cruise onboard YOKOSUKA (October, 2008). The locations of the

cores are shown in Fig. 1 and the bottom water oxygen concentrations in Table 1. The core locations were selected based on the intensity of OMZ, as the 500 m site experiences severe oxygen deficiency compared to 800 m, whereas the 1100 m site can be considered outside the intense OMZ. One core from each depth was selected for the analysis. The sediment cores were sectioned at 1 cm interval in the dark and stored at –20 °C until the analysis. Precautions were taken to minimize the degradation of pigments by light and temperature.

2.2. Phytoplankton pigment analysis

For phytoplankton pigment analyses, ~1 g freeze dried and homogenized samples were extracted with 3 ml 100% acetone in a glass centrifuge tube covered with aluminium foil. After sonication for 5 min in water bath containing ice, the extracts were kept at –20 °C over night. The extracts were centrifuged (5 min, 3000 rpm, T < 5 °C) and filtered using 0.2 µm PTFE syringe filters (Whatmann) and collected in amber coloured vials and kept at –20 °C in dark condition. The extract was concentrated near to dryness using nitrogen stream and made up to 500 µl with methanol just before HPLC analysis. Sample was analyzed using HPLC (Agilent Technologies) equipped with C-18 column (Supelco, 15 cm × 4.6 mm, 3 µm). Elution at a rate of 1.5 ml/min was performed using a linear gradient program of 43 min as follows: initially, 100% solvent A (80/20, methanol/0.05 M ammonium acetate) and switch over to 100% B (80/20, methanol/acetone) within 20 min. An isocratic hold of 20 min with solvent B for elution of all the major pigments then change to 100% A for 3 min. The injection volume was 50 µl. Pigments were detected using diode array detector (DAD) over a range between 300 and 800 nm. Absorption chromatograms were extracted for the wavelengths 450 nm for quantification of carotenoids and 665 nm for quantification of chlorophyll *a* and pheopigments. Pheophorbides were presented as total pheophorbide. The external standard calibration was done using pigment standards purchased from DHI, Denmark. All the chemicals used were HPLC grade from Merck, Germany.

2.3. CaCO₃, carbon and nitrogen content and their isotopic analysis

The inorganic carbon (CaCO₃) was analyzed using Coulometer (UIC Inc. Model 5014) attached with an acidification module following Bhushan et al. (2001). The analytical grade CaCO₃ was used as reference standard. Accuracy and precision achieved were within the range of ±2%. The δ¹³C and δ¹⁵N along with organic carbon (C_{org}) and nitrogen (N) content were measured using Isotopic Ratio Mass Spectrometer (Delta V Plus, Thermo) coupled with an Elemental Analyzer (EA-EUROVECTOR) in a continuous flow mode. Approximately 3 mg of decalcified, dried sediment samples were flash combusted in EA at 1050 °C. Evolved CO₂ and NO_x were passed through a reduction column containing copper at 670 °C, and were finally purified and separated with Gas Chromatograph at 50 °C before being introduced into the mass spectrometer. Calibration was done using laboratory standard (n-Caprioic acid, C₆H₁₅NO₂) as explained in Agnihotri et al. (2008). Analytical precision of <2% was achieved using international and in-house lab standards throughout the analysis.

3. Results

3.1. Sedimentary pigments

We have quantified nine pigments including chlorophyll *a* and its degradation products, pheophytin and pheophorbide. Though we found different allomers of pheophorbide, it was difficult to

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