



Organic matter degradation in surface sediments of the Changjiang estuary: Evidence from amino acids

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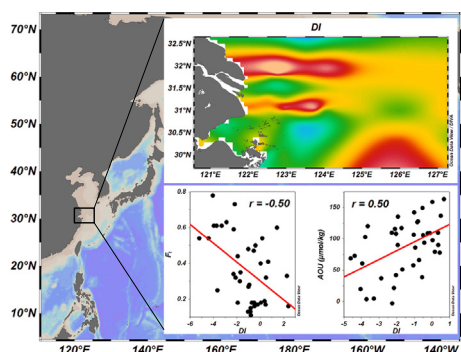
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HIGHLIGHTS

- An amino acids–based degradation index (*DI*) was mapped across the Changjiang Estuary.
- Sediments with more terrestrial material were generally more refractory.
- Organic matter was freshest in marine-sourced and silt-clay hypoxic sediments.
- *DI* was 39% associated with source material and bottom water oxygen combined.

GRAPHICAL ABSTRACT



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ABSTRACT

Organic matter degradation is a key component of the processes of carbon preservation and burial in seafloor sediments. The aim of this study was to explore organic matter degradation state within the open-shelf Changjiang Estuary of the East China Sea, using an amino acids–based degradation index (*DI*) in conjunction with information about organic matter source (marine versus terrestrial), bottom water oxygenation state, and sediment grain size. The relative molar percentages of 17 individual amino acids (characterized using principal component analysis) in surface sediments indicate that organic matter is degraded to varying extents across the estuary seabed. Sediments with $DI > 0$ (relatively labile) were found mostly within a coastal hypoxic area. Sediments of DI less than -1 (relatively refractory) were found near the Changjiang River mouth and the northern and southern parts of the central shelf. We consider *DI* to be a more reliable indicator of degradation than simple ratios of AAs. *DI* was inversely correlated with the proportion of terrestrial organic material (F_T) in the sediments, indicating that relatively fresh/labile organic matter was generally associated with marine sources. *DI* was significantly correlated with F_T and bottom water apparent oxygen utilization (AOU_{bot}) together. The parameter *DI* and the (labile) amino acid tyrosine were highest in hypoxic areas, suggesting the presence of relatively fresh organic matter, probably due to a combination of marine-source inputs and better preservation of organic matter in the silt and clay sediments of these areas (as compared to sandy sediments). Less degraded organic matter with high amino acids was also favorable to benthic animals. Overall, sedimentary estuarine organic matter was least degraded in areas characterized by marine sources of organic matter, low-oxygen conditions, and fine-grained sediments.

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1. Introduction

Marine surface sediments constitute an important carbon burial interface, where large amounts of organic carbon (OC) produced in upper waters are received, degraded (mostly by microorganisms) (Jørgensen, 2000), and finally buried. The consumption of oxygen in the course of organic matter degradation may strengthen hypoxia (Zhang et al., 2010), consequently amplifying ocean acidification (Hauri et al., 2009; Cai, 2011; Melzner et al., 2013) and accelerating the release of CO₂ to the atmosphere. On the shallower continental shelves, however, the factors influencing OM degradation are more complex. Fresh autochthonous estuarine organic matter mixing with highly degraded allochthonous terrestrial organic matter (Raymond and Bauer, 2001), strong horizontal oxygen gradients in estuaries (Hulthe et al., 1998; Vandewiele et al., 2009) and sediment grain size (Mayer, 1994), all these factors will affect the OM degradation states prominently. Understanding estuarine OM preservation and burial and its relation to carbon cycling requires the identification of differences in OM degradation state as well as the main factors or processes affecting OM in surface sediments.

Amino acids (AAs) are useful indicators of degradation in the marine environment (Cowie and Hedges, 1992; Dauwe et al., 1999; Lee et al., 2000). For example, intracellular amino acids present in cell plasma (glutamic acid, tyrosine, phenylalanine, and methionine) (Hecky et al., 1973) are more susceptible to degradation than are cell wall-associated amino acids (glycine, serine, alanine, and oysine). Similarly, the mole percentages of some non-protein amino acids, (e.g., β -alanine + γ -aminobutyric acid, β -Ala + γ -Aba) are elevated in sedimentary mixtures exposed to oxic degradation, because non-protein AAs are much less reactive than protein AAs. (Cowie and Hedges, 1992; Cowie et al., 1992; Dauwe and Middelburg, 1998; Suthhof et al., 2000).

The Changjiang Estuary of the East China Sea is a typical large river-dominated estuary, globally prominent among continental shelf ecosystems. Across the Changjiang Estuary shelf, fresh, labile (autochthonous) marine organic matter is mixed with degraded and refractory terrestrial organic matter to varying extents. Meanwhile, strong summer stratification of the water column in conjunction with the sinking and respiration of biogenic particles frequently results in hypoxic events (Li et al., 2002; Wei et al., 2007; Zhou et al., 2009; Qian et al., 2016). These variability in OM source and oxygen conditions would be expected to influence the bulk degradation state of the surface sediments. Previous studies in the region have used particulate and dissolved AAs to investigate the degradation of suspended particulate matter (Wu et al., 2007a; Zhu et al., 2014). Still, local controls on OM degradation state in the sediments (e.g., different OM sources or bottom-water oxygen conditions) remain unclear, as do their interrelationships.

In this study, we used amino acid pool compositions to quantify OM degradation state in the sediments of the Changjiang Estuary and established correlations of degradation state with marine versus terrestrial source contributions and bottom water oxygen utilization. We also compared different AA-based methods of quantifying organic matter degradation state or lability. The overarching aim was to provide a better understanding of sedimentary source and oxygen gradient controls on OM degradation in estuarine sediments.

2. Material and methods

2.1. Study area

The Changjiang Estuary (Fig. 1) is one of the world's most productive continental shelf areas. Annually, the Changjiang River (Yangtze River) delivers to the estuary a large sediment load of approximately 4.8×10^8 t (Milliman and Meade, 1983) and a freshwater discharge of approximately 14.8×10^7 m³ (Dai et al., 2014). Mixing of this eutrophic fresh water with the oligotrophic water of the Kuroshio Current gives

rise to strong cross-shelf gradients of salinity and nutrients (Zhang et al., 2007a). Marine biogenic particles resulting from surface-water primary production settle toward the seafloor, especially in the vicinity of the river plume, and are respired in the water column and surface sediments, resulting in oxygen consumption and frequent seasonal hypoxia (Li et al., 2002; Wei et al., 2007; Zhu et al., 2011; Zhu et al., 2016). The season for hypoxia events usually begins in early June and may persist as late as October (Zhu et al., 2016; Zhou et al., 2017).

2.2. Sampling methods

Samples of surface sediments (0–2 cm depth; $n = 149$) were collected at 149 stations during a research cruise in the Changjiang Estuary, 4 April to 17 May 2007 (Fig. 1). A steel grab sampler was used to recover the sediments, which were subsampled on deck and then frozen at -20 °C for later analysis.

Samples of suspended particulate matter ($n = 18$) were collected at 4 stations during 1–17 June 2009 (Fig. 1). Each sample of 500–2000 ml seawater was passed through a 47 mm diameter GF/F filter (pore size of 0.7 μ m) before being stored at -20 °C for later analysis. For chlorophyll *a* (Chl *a*) analyses, each sample of 100–250 ml seawater was passed through a GF/F filter, which was then stored at -20 °C for later processing.

Bottom waters (sampled at a height of 2 m above the seabed) were collected at 149 stations during 15 July to 10 September 2006 (Fig. 1). Seawater was collected into Niskin bottles attached to a CTD rosette, and the samples ($n = 149$) were later analyzed for dissolved oxygen content.

The three sample sets were collected during different time periods, but we consider them to be generally representative of typical summer conditions in our study area. In recent decades, summer hypoxia has typically occurred in the same general areas (Zhu et al., 2011). In addition, surface sediment geochemistry exhibits little seasonal variability due to low sedimentation rates in most parts of the estuary (Liu et al., 2006).

2.3. Sediment analyses: AAs, OC, TN, $\delta^{13}\text{C}$, F_p , and grain size

To quantify sedimentary total hydrolyzable amino acids (THAAs; $n = 47$) and HAs ($n = 47$), we dried and ground sediment subsamples before washing them with Milli-Q® water in a supersonic box for desalting. Aqueous hydrolyses were conducted in 6 N HCl under N₂ gas for 24 h at 110 °C. The hydrolysis mixture was dried and then dissolved in Milli-Q® water; the component amino acids were derived according to the Waters® AccQ-Tag™ method (Cohen and Michaud, 1993). The derivatives were determined with a Waters® 600E pump system (multisolute delivery system) and a Waters® 474 fluorescence detector; the THAA and HA data were processed with a Waters® Millennium®³² chromatography station. Results from duplicate samples of individual AAs showed the coefficient of variation to be <5.6%; for THAA, the coefficient of variation was 2.1%.

The external AA and HA standard solution (Sigma-Aldrich®) contained 17 individual amino acids: alanine (Ala), arginine (Arg), aspartic acid (Asp), cystine (Cys), glutamic acid (Glu), glycine (Gly), histidine (His), isoleucine (Iso), leucine (Leu), lysine (Lys), methionine (Met), phenylalanine (Phe), proline (Pro), serine (Ser), threonine (Thr), tyrosine (Tyr), and valine (Val). To this solution, the hexosamine standards galactosamine (Gal) and glucosamine (Gluco) were added, as were two non-protein amino acids, β -alanine (β -Ala) and γ -amino butyric acid (γ -Aba). The internal standard α -amino butyric acid (α -Aba) was also added for the determination of recovery rate, which was calculated to be between 92.1% and 103.7%.

After inorganic carbon was removed from the sediment samples (using 1 N HCl), concentrations of organic carbon ($n = 149$) and total nitrogen ($n = 149$) and ratios of stable carbon isotopes ($n = 149$) were determined with an isotope ratio mass spectrometer (Delta Plus

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