Pore water data from sediment cores collected from R/V Oceanus cruise OC1906A and R/V Sikuliaq cruise SKQ202016S off the coast of California in 2019 and 2020

Website: https://www.bco-dmo.org/dataset/959247

Data Type: Cruise Results

Version: 1

Version Date: 2025-04-16

Project

» Collaborative Research: Peptide Deamination as a Source of Refractory Dissolved Organic Matter in Marine Sediments (Peptide Deamination)

Contributors	Affiliation	Role
Burdige, David J.	Old Dominion University (ODU)	Principal Investigator
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Abstract

Sediment pore waters were determined in cores collected at several stations off the coast of California: at a site in Santa Barbara Basin ('SBB'; latitude: 34.223, Longitude: -119.986; water depth, 590 m); Catalina Basin ('Cat'; latitude: 33.301, longitude: -118.6; water depth, 1310 m); and two sites off the coast of central California south of Monterey Bay; site 'K' (latitude 35.375, longitude -121.501, water depth, 1000 m) and site 'D' (latitude 36.113, longitude: -122.186, water depth, 1440 m). The Cat cores were collected in June 2019, while the site K and D cores were collected in December 2020. Pore waters were collected either using rhizon samplers or by centrifugation of sectioned sediments. Pore water samples were analyzed for alkalinity on-board the ship, while other analyses were returned to the shore-based lab at Old Dominon University (ODU) where they were analyzed. These samples were collected as part of a larger project examining deaminated peptides and organic matter cycling in the sediments at these sites.

Table of Contents

- <u>Coverage</u>
- <u>Dataset Description</u>
 - Methods & Sampling
 - BCO-DMO Processing Description
- Data Files
- Related Publications
- Parameters
- <u>Instruments</u>
- <u>Deployments</u>
- Project Information
- <u>Funding</u>

Coverage

Location: Central California continental margin

Spatial Extent: N:36.1126 **E**:-118.6 **S**:33.3005 **W**:-122.1861

Temporal Extent: 2019-06-23 - 2020-12-09

Methods & Sampling

Field sampling methods:

Sediment cores were collected using a gravity corer and a multicorer on both cruises. Following recovery, the gravity cores were secured horizontally on the ship's deck and sampled from the bottom of the core upwards. This was done by sequentially removing 10-centimeter (cm) sediment intervals by cutting the core liner using a pipe cutter. Freshly exposed sediment was immediately subsampled using 3- to 60-milliliter (mL) push corers

made of plastic syringes with the tips removed. All subcores were immediately transferred to an N2 filled glove bag in a refrigerated van for further processing. Multi cores were transferred to a refrigerated van immediately upon recovery, and within 12 hours were either sampled using pre-cleaned Rhizon samplers (Seeberg-Elverfeldt et al., 2005) or extruded and sectioned in an N2 atmosphere at intervals of 0.5 to 2 cm depth over the depth of the core (generally 30 to 40 cm total length).

While it is possible to recover intact sediment-water interfaces using multi-corers, loss of surface sediments is typical during gravity coring, making it impossible to directly quantify absolute depths below the sediment-water interface in a gravity core. We therefore determined absolute depths of sediment sample intervals in gravity cores by aligning DIC, SO42-, NH4+, and porosity gravity core profiles to multicore profiles from the same site (Berelson et al., 2005; Iversen and Jørgensen, 1985; Komada et al., 2016).

Sediment aliquots from both gravity cores and sectioned multi cores were centrifuged at 6 degrees Celsius (°C) in polycarbonate tubes, and the supernatant was collected into all-polypropylene syringes with stainless steel needles. Rhizon samples were also collected in all poly-propylene syringes. All pore water samples were then filtered through disposable 0.2 micrometer (μ m) nylon filters with 0.7 μ m GF/F pre-filters (Whatman 6870-2502). The first 3 mL were discarded. To minimize the DOC blank, 100 mL of UV-irradiated deionized water (DIWUV) were pushed through each disposable filter prior to use.

Pore water samples for DIC concentration were placed in 2-mL serum vials without headspace, immediately crimp sealed, and refrigerated until analysis (Burdige and Homstead, 1994). Alkalinity samples were collected in 3-mL plastic syringes, sealed with 3-way stopcocks, and titrated on-board the ship within 24 hours. Titrated alkalinity samples were placed in snap cap vials and later used for sulfate and ammonium determinations.

Pore water samples for DOC concentration were acidified to pH < 2 with 6 N trace metal grade HCl and flame-sealed in pre-combusted glass ampules under a stream of UHP N2 gas and refrigerated.

Pore water samples for total dissolved sulfide (Σ H2S = [H2S] + [HS-] + [S2-]) analysis were fixed onboard ship by adding pore water to an N2-degassed solution containing 4 mL of 5 millimolar (mM) ZnCl2 and and 4 mL of 10 mM NaOH (Ingvorsen and Jørgensen, 1979) in a 10 mL serum bottle. At the basic pH of this "fixing" solution, all dissolved inorganic sulfide precipitates out as ZnS. The headspace was then degassed with N2 and the bottle was crimp sealed with plug-style stoppers and refrigerated. In SBB pore water samples near the sediment surface (upper 10 cm) where sulfide levels are low, 1 mL of pore water was added to the fixing solution, whereas for deeper samples (with higher sulfide levels) 0.1 mL pore water was added to the fixing solution. In station D and K pore water samples, 1-2 mL pore water was added to the fixing solution. Σ H2S was not determined in Cat pore water samples.

Bottom-water samples were collected from 10 meters (m) above the seafloor with a Go Flo bottle. All tools and parts used in all sampling were first cleaned with household dish soap, then acid rinsed (exclusive of metal parts). Plasticware was air dried.

Analytical methods:

Alkalinity samples were titrated on-board the ship by automated Gran titration (Burdige et al., 2010). pH values listed here are the initial pH values from the titrations and are expressed on the NBS scale. Concentrations of DIC and ammonium (SBB and Cat samples) were analyzed by flow injection analysis (Hall and Aller, 1992; Lustwerk and Burdige, 1995). Ammonium (stations D and K) was determined by the fluorometric technique of Holmes et al. (1999). Sulfate was determined by ion chromatography with conductivity detection (Komada et al., 2016). Σ H2S was determined spectrophotometrically using the methylene blue technique (Cline, 1969). All reagents were added directly to the serum bottle containing the ZnS suspension (see the section above for details).

Concentrations of DOC were determined by high-temperature combustion using a Shimadzu TOC-V total carbon analyzer (Burdige and Gardner, 1998). DOC consensus reference materials (D. Hansell, RSMAS) were run along with samples, and measured values agreed to within <10% with the reported consensus values.

BCO-DMO Processing Description

- Imported original file "pore water data.txt" into the BCO-DMO system.
- Flagged "nd" as a missing data value (missing data are empty/blank in the final csv file).
- Converted Date column to YYYY-mm-dd format.
- Added cruise ID column.
- Saved final file as "959247_v1_pore_water.csv".

Data Files

File

959247_v1_pore_water.csv(Comma Separated Values (.csv), 23.47 KB)

MD5:e1f85fa97bdb8d5d2e8b7287d92de8d2

Primary data file for dataset ID 959247, version 1

[table of contents | back to top]

Related Publications

Berelson, W. M., Prokopenko, M., Sansone, F. J., Graham, A. W., McManus, J., & Bernhard, J. M. (2005). Anaerobic diagenesis of silica and carbon in continental margin sediments: Discrete zones of TCO2 production. Geochimica et Cosmochimica Acta, 69(19), 4611–4629. doi:10.1016/j.gca.2005.05.011

Methods

Burdige, D. J., & Gardner, K. G. (1998). Molecular weight distribution of dissolved organic carbon in marine sediment pore waters. Marine Chemistry, 62(1-2), 45-64. https://doi.org/10.1016/s0304-4203(98)00035-8 https://doi.org/10.1016/s0304-4203(98)00035-8 Methods

Burdige, D. J., & Homstead, J. (1994). Fluxes of dissolved organic carbon from Chesapeake Bay sediments. Geochimica et Cosmochimica Acta, 58(16), 3407–3424. https://doi.org/10.1016/0016-7037(94)90095-7 *Methods*

Burdige, D. J., Hu, X., & Zimmerman, R. C. (2010). The widespread occurrence of coupled carbonate dissolution/reprecipitation in surface sediments on the Bahamas Bank. American Journal of Science, 310(6), 492–521. https://doi.org/10.2475/06.2010.03

Methods

Cline, J. D. (1969). Spectrophotometric Determination of Hydrogen Sulfide in Natural Waters. Limnology and Oceanography, 14(3), 454–458. doi: $\frac{10.4319}{0.1969.14.3.0454}$ Methods

Hall, P. . J., & Aller, R. C. (1992). Rapid, small-volume, flow injection analysis for total CO2, and NH4+ in marine and freshwaters. Limnology and Oceanography, 37(5), 1113–1119. doi:10.4319/lo.1992.37.5.1113

Methods

Holmes, R. M., Aminot, A., Kérouel, R., Hooker, B. A., & Peterson, B. J. (1999). A simple and precise method for measuring ammonium in marine and freshwater ecosystems. Canadian Journal of Fisheries and Aquatic Sciences, 56(10), 1801–1808. doi:10.1139/f99-128

Methods

Ingvorsen, K., & Jorgensen, B. B. (1979). Combined measurement of oxygen and sulfide in water samples. Limnology and Oceanography, 24(2), 390–393. doi:10.4319/lo.1979.24.2.0390

Methods

Iversen, N., & Jorgensen, B. B. (1985). Anaerobic methane oxidation rates at the sulfate-methane transition in marine sediments from Kattegat and Skagerrak (Denmark)1. Limnology and Oceanography, 30(5), 944–955. Portico. https://doi.org/10.4319/lo.1985.30.5.0944 Methods

Komada, T., Burdige, D. J., Li, H.-L., Magen, C., Chanton, J. P., & Cada, A. K. (2016). Organic matter cycling across the sulfate-methane transition zone of the Santa Barbara Basin, California Borderland. Geochimica et Cosmochimica Acta, 176, 259–278. doi:10.1016/j.gca.2015.12.022

Methods

Lustwerk, R. L., & Burdige, D. J. (1995). Elimination of dissolved sulfide interference in the flow injection determination of SCO2, by addition of molybdate. Limnology and Oceanography, 40(5), 1011–1012. doi:10.4319/lo.1995.40.5.1011

Methods

Seeberg-Elverfeldt, J., Schlüter, M., Feseker, T., & Kölling, M. (2005). Rhizon sampling of porewaters near the sediment-water interface of aquatic systems. Limnology and Oceanography: Methods, 3(8), 361–371. doi:10.4319/lom.2005.3.361

Methods

[table of contents | back to top]

Parameters

Parameter	Description	Units
Ship	Ship used: Oc = R/V Oceanus; Sk = R/V Sikuliaq	unitless
Cruise_ID	Cruise ID	unitless
St_ID	Station ID: SBB = Santa Barbara Basin; Cat = Catalina Basin; K = station K; D = station D	unitless
date	Date core was collected	unitless
lat	Station latitude	decimal degrees
long	Station longitude; negative values = West	decimal degrees
SA_ID	Individual sample ID	unitless
Core	Type of core used to collect the sample: H = hydrocast (bottom water samples); M = multi-core; G = gravity core	unitless
Samp	How the sample was collected: $GF = GO$ -Flo bottle (bottom water samples); $C =$ centrifugation; $R =$ rhizon samplers	unitless
Depth	Depth of the sediment sample	centimeters (cm)
err	half-depth of the sampling interval; no data for GF and R samples	centimeters (cm)
Alk	concentration of pore water alkalinity	millimolar (mM)
рН	pore water pH (NBS scale)	unitless
DIC	concentration of pore water dissolved inorganic carbon	millimolar (mM)
Sulfate	concentration of pore water sulfate	millimolar (mM)
Sulfide	concentration of pore water total dissolved H2S (?H2S)	micromolar (uM)
NH4	concentration of pore water ammonium	micromolar (uM)
DOC	concentration of pore water dissolved organic carbon	millimolar (mM)

Instruments

Dataset- specific Instrument Name	Agilent model 8453 UV-Vis Spectrophotometer
Generic Instrument Name	Agilent 8453 UV-visible spectrophotometer
Dataset- specific Description	used to measure total dissolved sulfide
Generic Instrument Description	The Agilent 8453 spectrophotometer is a laboratory optical instrument for chemical analysis to extract spectral information in the ultraviolet (UV) and visible light. The instrument radiates a single light beam by optically combining two source lamps: a deuterium-discharge lamp for the UV wavelength range and a tungsten lamp for the visible and short wave near-infrared (SWNIR) wavelength range. The beam passes through the sample, is focused and dispersed within the spectrograph lens, slit and grating, and reaches the diode array in the form of a spectral image. The diode array samples a wavelength range of 190 to 1100 nm at a mean sampling interval of 0.9 nm. The nominal spectral slit width is 1 nm and the stray light is less than 0.03%.

Dataset-specific Instrument Name	Centrifuge
Generic Instrument Name	Centrifuge
Generic Instrument Description	A machine with a rapidly rotating container that applies centrifugal force to its contents, typically to separate fluids of different densities (e.g., cream from milk) or liquids from solids.

Dataset- specific Instrument Name	Dionex CDM-II conductivity detector
Generic Instrument Name	Conductivity Meter
Dataset- specific Description	used to measure dissolved ammonium and dissolved inorganic carbon
Generic Instrument Description	Conductivity Meter - An electrical conductivity meter (EC meter) measures the electrical conductivity in a solution. Commonly used in hydroponics, aquaculture and freshwater systems to monitor the amount of nutrients, salts or impurities in the water.

Dataset- specific Instrument Name	GO-Flo sampling bottle
Generic Instrument Name	GO-FLO Bottle
Dataset- specific Description	used to collect bottom water samples
	GO-FLO bottle cast used to collect water samples for pigment, nutrient, plankton, etc. The GO-FLO sampling bottle is specially designed to avoid sample contamination at the surface, internal spring contamination, loss of sample on deck (internal seals), and exchange of water from different depths.

Dataset- specific Instrument Name	"Big Bertha" gravity corer (built at Oregon State University)
Generic Instrument Name	Gravity Corer
Dataset- specific Description	used to collect gravity cores
Generic Instrument Description	The gravity corer allows researchers to sample sediment layers at the bottom of lakes or oceans. The coring device is deployed from the ship and gravity carries it to the seafloor. (http://www.whoi.edu/instruments/viewInstrument.do?id=1079).

Dataset- specific Instrument Name	Ocean Instruments Mult-Corer
Generic Instrument Name	Multi Corer
Dataset- specific Description	used to collect sediment cores
Generic Instrument Description	The Multi Corer is a benthic coring device used to collect multiple, simultaneous, undisturbed sediment/water samples from the seafloor. Multiple coring tubes with varying sampling capacity depending on tube dimensions are mounted in a frame designed to sample the deep ocean seafloor. For more information, see Barnett et al. (1984) in Oceanologica Acta, 7, pp. 399-408.

Dataset-specific Instrument Name	Rhizon samplers
Generic Instrument Name	Sediment Porewater Sampler
Dataset-specific Description	used for collecting pore water samples from multicore extrusions and other processes
Generic Instrument Description	A device that collects samples of pore water from various horizons below the seabed.

Dataset-specific Instrument Name	Shimadzu TOC-V total carbon analyzer
Generic Instrument Name	Shimadzu TOC-V Analyzer
Dataset-specific Description	used to measure dissolved organic carbon
Generic Instrument Description	A Shimadzu TOC-V Analyzer measures DOC by high temperature combustion method.

Dataset- specific Instrument Name	Thermo-Fisher Dionex ICS-5000 ion chromatograph
Generic Instrument Name	Thermo Fisher Scientific Dionex ICS-5000 ion chromatography (IC) system
Dataset- specific Description	used to measure sulfate
	The Thermo Fisher Scientific Dionex ICS-5000 ion chromatography (IC) system is an ion chromatography system that offers a full range of reagent-free components. This instrument can be configured to use single or dual pumps. The single-channel Dionex ICS-5000 can be configured to run capillary, microbore or standard bore IC applications. A dual Dionex ICS-5000 system can be configured with any combination of these applications. This system uses an eluent generator (EG) to generate high purity acid or base eluents from deionized water, in the amount and concentration needed for sample analysis, configurable for single or dual channel operation. Eluent regeneration may also be used without an EG - eluent regeneration uses the suppressor to reconstitute the starting eluent, allowing use of a single 4-liter bottle of eluent for up to four weeks. An eluent organizer (EO) module is used to contain eluent spills and leaks. The ICS-5000 detector/chromatography module (DC) can accommodate components for two channels, plumbed either serially or in parallel, in a temperature-controlled environment. Available DC components include conductivity detectors, electrochemical detectors, injection valves, switching valves, guard and separator columns, suppressors, and Dionex IC cubes or ICS-5000 Automation Manager. Detectors outside of the DC include a Dionex ICS Series Photodiode Array Detector (PDA); Dionex ICS Series Variable Wavelength Detector (VWD); MSQ Plus Mass Spectrometer.

Dataset-specific Instrument Name	Metrohm automatic titrator (model 785 DMP Titrino)
Generic Instrument Name	Titrator
Dataset-specific Description	used to measure alkalinity and initial pH
Generic Instrument Description	Titrators are instruments that incrementally add quantified aliquots of a reagent to a sample until the end-point of a chemical reaction is reached.

[table of contents | back to top]

Deployments

OC1906A

Website	https://www.bco-dmo.org/deployment/914972	
Platform	R/V Oceanus	
Start Date	2019-06-20	
End Date	2023-07-03	
Description	See more information at R2R: https://www.rvdata.us/search/cruise/OC1906A	

SKQ202016S

5/1920203		
Website	https://www.bco-dmo.org/deployment/915307	
Platform	R/V Sikuliaq	
Start Date	2020-12-01	
End Date	2020-12-12	
Description	See more information at R2R: https://www.rvdata.us/search/cruise/SKQ202016S	

[table of contents | back to top]

Project Information

Collaborative Research: Peptide Deamination as a Source of Refractory Dissolved Organic Matter in Marine Sediments (Peptide Deamination)

Coverage: California Borderland

NSF Award Abstract:

Dissolved organic matter (DOM) in the ocean is one of the largest carbon reservoirs on Earth. Much of this DOM is highly resistant to degradation (refractory) and aged, but the nature and reasons behind the accumulation of refractory DOM in the ocean is one of the unresolved mysteries of the marine carbon cycle. While marine sediments have been shown to be a globally important source of DOM to the ocean, the connection between sediment DOM dynamics and the oceanic DOM cycle remains elusive, because information is lacking on the molecular composition and reactivity of pore water DOM. To fill this knowledge gap, this project will address the question of how refractory DOM is produced in sediments and the fate of benthic DOM in the water column. The research will focus on the relationship between protein/peptide dynamics and sediment DOM cycling, examining peptide deamination as an important pathway for the production of refractory and 14C-depleted DOM in continental margin sediments. These objectives will be met through a combination of geochemical profiling of sediment cores collected across a range of redox conditions, and longterm sediment incubation studies conducted under controlled laboratory conditions. At the heart of this proposed work is structural elucidation and quantification of intact and deaminated peptides in pore-water DOM using state-of-the-art analytical techniques. The study will help better understand how the present-day carbon cycle operates, as well as how it may respond in the future. The proposed work will integrate research and education using several approaches. All PIs routinely integrate their research into their classes, which range from introductory-undergraduate to advanced-graduate courses and will continue to do so here. All three PIs are also committed to engaging women and underrepresented minority students.

Marine sediments are a globally important source of dissolved organic matter (DOM) to the ocean. However, the connection between sediment DOM dynamics and the oceanic DOM cycle remains elusive because information about the molecular composition and reactivity of pore water DOM is lacking. To help fill this knowledge gap, this project will address the question of how refractory DOM is produced in sediments and the fate of the benthic DOM flux in the water column. The proposed study explores a novel and potentially transformative idea that deamination of peptides in sediments is a source of refractory and 14C-depleted DOM in seawater. This idea is consistent not only with the fact that the majority of seawater dissolved organic nitrogen occurs in amide form, but also with recent reports about the widespread occurrence of nitrogen-bearing formulas in deep-sea refractory DOM. The central hypothesis will be tested through a unique blend of bottom-up (molecular level DOM analyses) and top-down (bulk-level elemental and isotopic analyses, and

numerical modeling) approaches. This work will involve a combination of geochemical profiling of sediment cores collected across a range of redox conditions, and long-term sediment incubation studies conducted under controlled laboratory conditions. At the heart of the proposed work is structural elucidation and quantification of intact and deaminated peptides in pore-water DOM using a state-of-the-art liquid chromatography-mass spectrometry system (ultra-high performance liquid chromatography coupled to an Orbitrap Fusion Tribrid Mass Spectrometer), which is expected to provide an unprecedented wealth of molecular-level information about pore water DOM. The proposed work will lead to an improved mechanistic understanding of organic matter decomposition and benthic DOM cycling and shed light on the connections between the modern-day oceanic and sedimentary carbon and nitrogen cycles as they relate to the formation of refractory DOM.

This award reflects NSF's statutory mission and has been deemed worthy of support through evaluation using the Foundation's intellectual merit and broader impacts review criteria.

[table of contents | back to top]

Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1756669

[table of contents | back to top]