1H NMR spectra of whole porewater dissolved organic matter (DOM) samples from California margin sediments sampled in 2020 onboard the R/V Sikuliaq

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

Contributors	Affiliation	Role
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Abstract
1H NMR spectra of whole dissolved organic matter (DOM) samples are reported for StaD and StaK, located in the upper slope offshore central California. Sediment and porewater sampling were done in Dec 2020 onboard the R/V Sikuliaq. Sediment samples were retrieved using multi-cores and gravity cores operated by the Oregon State University Marine Rock and Sediment Sampling Group. Porewater samples were extracted from the sediment within hours of core retrieval in a refrigerated van. Rhizon samplers were used to collect porewater from multicores, whereas centrifugation was used for porewater collection from gravity cores. Upon collection, porewater samples were immediately poisoned with mercuric chloride and flame-sealed in glass ampules under a stream of ultra-high-purity nitrogen gas and refrigerated until analysis. Reported here are 1H NMR spectra of whole DOM samples, UV-irradiated deionized water blanks collected on the ship, and UV-irradiated deionized water pulled through Rhizon samplers (all 0.2-micron filtered).

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Coverage

Location: Station D sediments, 36.19/-122.31, 0-275 cm Station K sediments, 35.63/-121.84, 0-285 cm
Spatial Extent: **N**:36.1875 **E**:-121.83534 **S**:35.62588 **W**:-122.31074
Temporal Extent: 2020-12-04 - 2020-12-09

Methods & Sampling

Porewater samples from gravity cores were collected as described in Komada et al. (2016) Geochim.

Cosmochim. Acta, 176:259-278. Briefly, sediment cores were sectioned within hours of retrieval, then centrifuged, filtered (0.2 micron pore size). Porewater samples from multicores were extracted using Rhizon samplers (Seeberg-Elverfeldt et al., Limnol. Oceanogr. Meth., 8:361-371, 2005) to prevent contamination from squashed benthic macrofauna during centrifugation. Samples from multicores (MC) were retrieved using Rhizon samplers (5 cm, 0.15 micron pore size). Samples from BB gravity cores were collected by centrifugation, then filtered through 0.2 miron filters. Samples were immediately poisoned with HgCl2 and flame-sealed under a stream of high-purity nitrogen gas and refrigerated until analysis.

Blank samples: MQ_blank samples = UV-irradiated MQ water collected on the ship in parallel with porewater samples; Rhizon_blank samples = UV-irradiated MQ water collected on in the laboratory after passing it through a Rhizon sampler.

1H NMR spectra were acquired in 5-mm O.D. thin-wall precision NMR tube (Bel-Art Wilmad part #528-PP-7-5) in 5% D2O (Sigma Aldrich part #151882) using a Bruker Avance NEO 500 MHz instrument with Prodigy cryoprobe using W5-WATERGATE solvent suppression method (Lam and Simpson, Analyst, 133:263-269, 2008).

Data Processing Description

NMR spectra were normalized by the total area under the spectra after removing the residual water peak (4.2-5.0 ppm).

BCO-DMO Curation Notes

* Converted original data that was in wide format to long format. Both files are part of this dataset.
* Added sampling latitude and logitude to dataset
* Adjusted parameter names to comply with database requirements (no periods, spaces or special characters)

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Data Files

File
964221_v1_nmr.csv (Comma Separated Values (.csv), 83.23 MB) MD5:458728dfc884f5daa5193fe63779d99a Primary data file for dataset ID 964221, version 1

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Supplemental Files

File
Porewater DOM 1H NMR StaD_StaK filename: porewater DOM 1H NMR StaD_StaK.xlsx (Microsoft Excel, 13.69 MB) MD5:66adaef639efb6bb592e6c8560baedcb NMR spectra from Stations D and K in original wide format.

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Related Publications

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

Lam, B., & Simpson, A. J. (2008). Direct 1H NMR spectroscopy of dissolved organic matter in natural waters. The Analyst, 133(2), 263–269. https://doi.org/10.1039/b713457f https://doi.org/10.1039/B713457F
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

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Parameters

Parameter	Description	Units
station	D and K represent Station D (36.1875 latitude, -122.31074 longitude) and Station K (35.62588 latitude, -121.83534 longitude). Further details can be found in Reimers et al. (1992) Global Biogeochem. Cycles, 6:199-224.	unitless
latitude	Latitude of sampling station, south is negative	decimal degrees
longitude	Longitude of sampling station, west is negative	decimal degrees
sampling_date	Sampling date	unitless
sample_type	Porewater samples = whole samples without pre-extraction or concentration. Blanks = test samples processed in similar manner to porewater samples to assess contamination.	unitless
core_type	MC = multicores. BB = Big Bertha gravity core operated by Oregon State University Marine Rock and Sediment Sampling Group	unitless
sample_ID	sample ID: combination of station, core type, analysis date = last 6 digits of sample ID mmddyy)	unitless
sediment_depth	Depth of sediment in core	centimeters (cm)
chemical_shift	1H chemical shift relative to tetramethylsilane	parts per million (ppm)
nmr_value	Area-normalized intensity	unitless

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Instruments

Dataset-specific Instrument Name	
Generic Instrument Name	Gravity Corer
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. From: http://www.whoi.edu/instruments/viewInstrument.do?id=1079

Dataset-specific Instrument Name	
Generic Instrument Name	Multi Corer
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	Bruker Avance NEO 500 MHz
Generic Instrument Name	Nuclear Magnetic Resonance Spectrometers
Dataset-specific Description	Bruker Avance NEO 500 MHz instrument with Prodigy cryoprobe using W5-WATERGATE solvent suppression method
Generic Instrument Description	Instruments that identify and quantify magnetically active chemical entities by subjecting a sample to orthogonal magnetic and electrical fields.

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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 long-term 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.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1756686

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