DOC and TDN concentrations & phenolic content from exudation experiments in outdoor tanks with Sargassum samples collected off the coast of Bermuda and in the Sargasso Sea in 2016

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Project
» Collaborative Research: Phlorotannins - An Important Source of Marine Chromophoric Dissolved Organic Matter? (Sargassum DOM)

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Abstract
Dissolved inorganic carbon (DOC), total dissolved nitrogen (TDN) concentrations, and phenolic content from exudation experiments in outdoor tanks with Sargassum samples collected aboard the R/V Henry Stommel off the coast of Bermuda and R/V Hugh.R. Sharp in the Sargasso Sea in 2016. This dataset includes formula assignments for outdoor exudation experiments under natural (sunlight) conditions (n = 4). These data were published in Powers et al. (2019).

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Coverage

Location: Bermuda and Sargasso Sea
Temporal Extent: 2016-06 - 2016-09

Dataset Description

See "Related Datasets" section for other datasets from these exudation experiments.

SPE-DOC = Solid-phase extracted dissolved organic carbon.
DOC = Dissolved inorganic carbon

TDN = Total Dissolved Nitrogen

Methods & Sampling

Sampling and analytical procedures:

Sargassum (urn:lsid:marinespecies.org:taxname:144132) was collected during two sampling events in July and in late September/early October 2016, described in detail in a previous study (Powers et al., 2019, Global Biogeochemical Sciences (GBC), *33*(11), 1423-1439). Briefly, samples were collected in July 2016 aboard the R/V *Hugh R. Sharp in* the Sargasso Sea, and were housed onboard in a tank (< 3 days) with continuously flowing seawater before it was transported to the Chesapeake Biological Laboratory (CBL) for exudation experiments. **These are exudation experiments are referred to as CBL exudation experiments.** *Sargassum* samples collected in early fall 2016 aboard the R/V *Henry Stommel*, 9 km off the coast of Bermuda, were used in outdoor exudation experiments. **These exudation experiments are referred to as BDA exudation experiments.**

Solid phase extraction (SPE). At the end of all incubation experiments that typically lasted 24 to 48 h, tank water housing *Sargassum* was filtered through pre-combusted Whatman 0.7 µm GF/F glass fiber filters, acidified to pH 2 using concentrated HCl and solid-phase extracted using Agilent Bond Elut PPL cartridges. Our solid phase extraction (SPE) technique utilized 10 g custom-packed cartridges that were activated with ultrapure methanol and rinsed with ultrapure 0.1% formic acid water prior to extraction. After extraction, cartridges were rinsed with 0.1% formic acid water and dried. SPE-DOM was eluted in 30 mL ultrapure MeOH and DOC extraction efficiency ranged from 40 – 60%. However, extraction efficiency was always low (~30%) for extractions of DOM leached from dried material or *Sargassum* incubated with no temperature control. Methanolic extracts were stored at -20 °C until use.

DOC and TDN measurements for SPE-DOM. Methanolic SPE extracts were brought to room temperature; subsamples were pipetted into combusted glass vials and dried under a steady stream of ultrapure N2 gas. Dried extracts were re-dissolved in a known volume of ultrapure water and acidified to pH 2 using concentrated HCl (Sigma Aldrich 32 %, pura). These reconstituted SPE-DOM samples were analyzed for dissolved organic carbon (DOC) and total dissolved nitrogen (TDN) concentrations using a Shimadzu TOC-V. Ultrapure water was used as a DOC/TDN blank and potassium hydrogen phthalate and potassium nitrate were used for DOC and TDN standards, respectively. These data are also reported in (Powers et al., 2019, GBC). DOC concentrations were corrected for sample volume extracted, methanol elution volume, dried methanol extract volume and ultrapure water volume. Corrected DOC values (SPE-DOC) were compared to DOC values for filtered water samples collected at the same time point before extraction (DOCsample). DOC extraction efficiency was calculated as 100x[SPE-DOC]/[DOCsample].

Phenol Concentrations. Phenolic content in SPE-DOM was determined using Folin-Ciocalteu method for the determination of phenolic content in solid-phase extracted marine DOM (Takeda et al. 2013, Marine Chemistry, *157*: 208-215). Briefly, SPE-DOM samples were created with 0.2 mL of each MeOH extract that dried completely under N2 and re-dissolved in 20 mL Milli-Q water, sonicated for 5 min, and 0.2 µm filtered (Whatman 25 mm GD/X syringe filters). 3.6 mL of each sample was transferred to a combusted borosilicate vial containing 0.4 mL 1 M NaOH (J.T. Baker) and 0.2 mL Folin-Ciocalteu phenol reagent (Sigma-Aldrich), and reacted for 30 min at room temperature. Subsequently, 4 mL 2 M Na2CO3 (Fisher) and 1.8 mL Milli-Q water was added to the reaction mixture, the color was developed for 1 h in an oven at 40°C and the mixture's absorbance was monitored in a 1 cm quartz spectrophotometric cell at 720 nm using a Horiba Aqualog. Phenol concentrations were determined by a calibration curve of phloroglucinol (Aldrich) solutions ranging from 0 to 50 µM that were reacted in the same way as the samples. Thus, phenol concentrations reported here are reported as phloroglucinol equivalents. The unreacted SPE-DOM sample was analyzed for DOC concentration, as above. Phenolic content was simply determined by dividing the phenol content (as µM phloroglucinol equivalents) by the SPE-DOM concentration (µM or mg) and reported as %phenolic content (100×[phenol content]µM/[DOC]µM) or as mol kg-1 C ([phenol content]µM/[DOC]mgC/L).

BCO-DMO Processing Description

Data from submitted file "Sargassum_Exudation_SPEDOM_DOC_phenols.xls" was imported into the BCO-DMO data system for this dataset. Values "NA" and "NaN" were imported as missing data values.
** Missing data values are displayed differently based on the file format you download. They are blank in csv

files, "NaN" in MatLab files, etc.

* Metadata for this dataset was extracted from file "DATASET_Sargassum_DOC_TDN_phenolic_content.rtf"

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Related Publications

Powers, L. C., Hertkorn, N., McDonald, N., Schmitt-Kopplin, P., Del Vecchio, R., Blough, N. V., & Gonsior, M. (2019). Sargassum sp. Act as a Large Regional Source of Marine Dissolved Organic Carbon and Polyphenols. Global Biogeochemical Cycles, 33(11), 1423–1439. Portico. https://doi.org/10.1029/2019gb006225 https://doi.org/10.1029/2019GB006225
Results

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Related Datasets

IsRelatedTo

Gonsior, M., Del Vecchio, R., Blough, N. V., Powers, L. (2024) **FT-ICR MS data from exudation experiments in outdoor tanks with Sargassum samples collected off the coast of Bermuda and in the Sargasso Sea in 2016.** Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 1) Version Date 2024-09-25 http://lod.bco-dmo.org/id/dataset/938799 [view at BCO-DMO]
Relationship Description: These datasets all utilized samples from the same outdoor exudation experiments.

Gonsior, M., Del Vecchio, R., Blough, N. V., Powers, L. (2024) **Fluorescence spectra from exudation experiments in outdoor tanks with Sargassum samples collected off the coast of Bermuda and in the Sargasso Sea in 2016.** Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 1) Version Date 2024-09-25 http://lod.bco-dmo.org/id/dataset/938831 [view at BCO-DMO]
Relationship Description: These datasets all utilized samples from the same outdoor exudation experiments.

Gonsior, M., Del Vecchio, R., Blough, N. V., Powers, L. (2024) **Sargassum DOM optical properties tested for photodegradation rates and pH dependence from experiments conducted in outdoor tanks with Sargassum samples collected off the coast of Bermuda and in the Sargasso Sea in 2016.** Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 1) Version Date 2024-09-25 http://lod.bco-dmo.org/id/dataset/938807 [view at BCO-DMO]
Relationship Description: These datasets all utilized samples from the same outdoor exudation experiments.

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Parameters

Parameter	Description	Units
EXP	Exudation experiment type	unitless
description	experimental details (time/treatment)	unitless
SPE_DOC_mg_L	Solid-phase extracted dissolved organic carbon (SPE-DOC) concentration (mg/L)	milligrams per Liter (mg/L)
SPE_DOC_uM	Solid-phase extracted dissolved organic carbon (SPE-DOC) concentration (uM)	micromolar (uM)
extraction_efficiency_percent	Dissolved Organic Carbon (DOC) extraction efficiency	percent(%)
phenol_uM_PG	Phenol in phloroglucinol equivalents (uM). See methodology for more details.	micromolar (uM)
phenol_umol_mg	Phenol/([SPE-DOC] mg/L). See methodology for more details.	micromoles per gram (umol/g)
percent_phenol	Phenol/([SPE-DOC] µM). See methodology for more details.	percent (%)

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Instruments

Dataset-specific Instrument Name	
Generic Instrument Name	Shimadzu TOC-V Analyzer
Dataset-specific Description	Shimadzu TOC-V (DOC and TDN concentrations)
Generic Instrument Description	A Shimadzu TOC-V Analyzer measures DOC by high temperature combustion method.

Dataset-specific Instrument Name	Horiba Aqualog spectrofluorometer
Generic Instrument Name	Spectrometer
Dataset-specific Description	Horiba Aqualog spectrofluorometer (phenolic content)
Generic Instrument Description	A spectrometer is an optical instrument used to measure properties of light over a specific portion of the electromagnetic spectrum.

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Project Information

Collaborative Research: Phlorotannins - An Important Source of Marine Chromophoric Dissolved Organic Matter? (Sargassum DOM)

Coverage: Mid-Atlantic Bight (July 2016), Sargasso Sea (July and September 2016), Coastal Bermuda (September/October 2016) and Coastal Puerto Rico (Laguna Grande, Fajardo; Las Croabas, Fajardo; Salinas; May/June 2018)

NSF Award Abstract:

Chromophoric dissolved organic matter (CDOM), the sunlight absorbing components in filtered water, is important in the study of marine and freshwater ecosystems as it can be used to trace the mixing of surface waters, as a proxy for carbon cycles, and other biogeochemical processes. Although its importance in ocean studies has been firmly established over the last several decades, sources and structural composition of CDOM within the oceans remains unclear and continues to be a subject of debate. Sargassum, a brown alga, is widely distributed in temperate and subtropical marine waters and may be important source of CDOM to the Sargasso Sea and Gulf of Mexico where Sargassum is abundant. This project will investigate the contribution of macro brown algae-derived compounds to the marine CDOM pool. Results from this study will have implications for the marine carbon cycle and satellite remote sensing of ocean color to assess mixing of surface water masses and biogeochemical processes. The project will provide educational opportunities for a postdoctoral scholar, summertime undergraduate internships (through a local NSF-sponsored Research Experiences for Undergraduates (REU) program), and workshop and research opportunities for local high schools students.

Sources of marine CDOM remain debatable and a comprehensive understanding of its origins, distribution and fate have been difficult. Marine CDOM, and in particular the "humic-like" component, have been suggested to originate from terrestrial sources, primarily lignins. However, recent evidence indicates that the exudation of phlorotannins produced by macro brown algae may contribute significantly to the marine CDOM pool. Phlorotannins, a class of polyphenols that are only found in, and continuously exuded by macro brown algae such as Sargassum, strongly absorb ultraviolet light and may have been underestimated in their contribution to the marine CDOM pool within certain geographic locales. Upon partial oxidation, light absorption by these specific compounds extends into longer wavelengths in the visible creating an absorption spectrum similar to that of lignin. These phlorotannins and their transformation products absorb light that might explain in part the "humic-like" signatures observed in open ocean environments. This study aims to characterize the optical properties and molecular composition of Sargassum-derived CDOM including its aerobic oxidation and photochemical behavior, as well as quantify Sargassum-derived CDOM to better estimate its possible contribution to the CDOM pool in the Sargasso Sea and Gulf of Mexico.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1536888
NSF Division of Ocean Sciences (NSF OCE)	OCE-1536927

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