Biogeochemistry of soils, sediments, and surface waters across the upland to wetland gradient of coastal interfaces

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Abstract

Transferable and mechanistic understanding of cross-scale interactions is necessary to predict how coastal systems respond to global change. Cohesive datasets across geographically distributed sites can enable a mechanistic understanding of coastal ecosystem control points and examine how geographically transferable this knowledge is. To address the above research objectives, data were collected by the EXploration of Coastal Hydrobiogeochemistry Across a Network of Gradients and Experiments (EXCHANGE) Consortium– a regionally distributed network of researchers that collaborated on experimental design, methodology, collection,

analysis, and publication. The EXCHANGE Consortium collected samples from 52 coastal terrestrial-aquatic interfaces (TAIs) during Fall of 2021. At each site, samples collected include soils from across a transverse elevation gradient (i.e., coastal upland forest, transitional forest, and wetland soils), surface waters, and nearshore sediments. The data described herein aims to characterize the baseline distribution and chemical forms of carbon, nutrients, iron redox and mineralogy across research sites in the Great Lakes and Mid-Atlantic regions (Chesapeake and Delaware Bays) of the continental USA. This first campaign measures surface water quality parameters (e.g., conductivity, pH, ORP, alkalinity, and total suspended solids); bulk geochemical parameters on water, soil, and sediment samples (e.g., carbon, nutrient, and ionic concentration and composition); and physicochemical parameters of sediment and soil (soil pH, conductivity, bulk density, water retention). Future campaigns will focus on building off these baseline datasets to enable a mechanistic understanding of coastal ecosystem biogeochemical control points.

Measurement(s)	water quality; physical properties; bulk nutrients; organic matter chemistry; redox chemistry
Sample Characteristic-Environment	Terrestrial-aquatic interface Upland forest, Wetland, Open water, Benthic
Sample Characteristic-Location	Great Lakes Region, Mid-Atlantic Region, Chesapeake Bay, Delaware Bay

Background & Summary

The structure and function of coastal ecosystems vary considerably across relatively small spatial scales, resulting in dynamic hydrological and biogeochemical behaviors along the gradient of coastal upland, wetland, and surface water environments [1,2]. Insight into drivers of spatial heterogeneity can be elucidated by linking biogeochemical data with ecosystem properties [3,4], enabling mechanistic understanding, model validation, and improved uncertainty constraints of coastal ecosystems [1,5].

Open access and interoperable coastal biogeochemical datasets are needed to predict how coastal systems will respond to global change [3,6]. The Great Lakes and Mid-Atlantic regions host a wealth of long-term monitoring programs such as the National Estuarine Research Reserve [7], the Great Lakes Wetland Monitoring Program [8], and the Chesapeake Bay Program [9], among others. However, the synthesis of existing datastreams across traditional ecosystem boundaries is still relatively sparse [1,5]. Here, we describe datasets collected as part of EXCHANGE Campaign 1 (EC1), which establishes a baseline understanding of the chemical forms and distribution of carbon and nutrients across coastal terrestrial-aquatic interface (TAI) research sites in the Great Lakes and Mid-Atlantic regions (Chesapeake and Delaware Bays) of the continental USA. EXCHANGE adds to the existing efforts in these regions by developing a consortium of regional researchers interested in exchanging knowledge and information with a molecular level focus that spans upland to aquatic domains.

During the Fall of 2021, the EXCHANGE Consortium collected samples from 52 coastal TAIs. At each site, samples collected include soils from across a transverse elevation gradient (i.e., coastal upland forest, transitional forest, and wetland soils), surface waters, and nearshore sediments (Figure 1). EC1 samples were analyzed for bulk geochemical parameters, bulk physicochemical parameters, organic matter characteristics, and redox-sensitive elements. These datasets can be utilized to assess physicochemical drivers of spatial variations in organic matter cycling across coastal TAIs, and to enable a cross-system transferable and mechanistic understanding of coastal ecosystem biogeochemical control points.

Methods

1. *Sampling and Processing*

1.1 Sampling Design

The experimental design of EC1 was developed via workshops (following open science principles [10]) from conception to data analysis and publication. Coastal researchers gathered virtually to design a spatially distributed sampling campaign across Great Lakes and Mid-Atlantic regions (Figure 1). The EC1 consortium collected surface waters, soils, sediments and site level metadata using standardized sampling kits. Following sample collection, all sample kits were shipped to the Marine and Coastal Research Laboratory (Sequim, WA), part of Pacific Northwest National Laboratory.

1.2 Site Metadata

At each site, the EXCHANGE consortium collected standardized site metadata, such as latitude, longitude, and type of water system (e.g., estuary, lake). Additional site metadata, such as elevation and soil type, were extracted from publicly available databases (e.g., GoogleEarth) using site coordinates.

1.3 Surface Waters

Field-filtered water, using 0.22 µm Sterivex syringe filters, was collected in vials for dissolved organic carbon (DOC), total dissolved nitrogen (TDN), common dissolved ions, stable water isotopes, and several organic matter characterization methods (e.g., colored dissolved organic matter (CDOM)). Samples were filtered into vials in the field and preserved by freezing or storing at 4˚C until analyzed, depending on the analyte (Table 1). A 125 mL amber HDPE bottle of unfiltered water was collected with no headspace for water pH, oxygen-reduction potential (ORP), alkalinity, and conductivity measurements. Unfiltered surface water samples were also collected in 1L acid cleaned HDPE brown bottles for total suspended solids and filtered to 0.2 µm in the lab, within 48 hours of collection. Lab-filtered 1L grab samples were extracted for several organic matter characterization methods (e.g., high-resolution mass spectrometry) using standard solid phase extraction (SPE) procedures [11] . The filtered samples were stored at 4˚C until SPEs were completed, within 2 weeks of sample collection. One liter of sample was passed through a 6 mL / 1 g PPL SPE cartridge (Agilent PPL) after being acidified to a pH of 2, 24 hours before extraction. Samples were then eluted in LC-MS grade methanol, and were stored at -20˚C until analysis. Common dissolved ions and stable water isotopes data will be included in future versions of the accompanying data package. Additional analysis beyond those reported herein will be performed on archived waters or SPE extracts, and appended to future versions of the data package [12].

1.4 Soils

Surface soils (top 5 cm of soil profile) were collected from the three transect locations (upland, transition, and wetland) from each site. Soils were collected as intact cores (using HYPROP sampling rings, 5 cm diameter x 5 cm depth) and as surface grab samples (using 2.5 oz plastic (clear polypropylene) jars and plastic bags). The intact cores were refrigerated at 4 ˚C upon arrival to the laboratory. Subsamples of grab samples were either immediately processed, frozen (-20 ˚C), or refrigerated, based on the analyses planned (Table 1). Frozen grab samples were freeze-dried and sieved to 5.6 mm before additional analyses. Water retention curves, particle size analysis, and X-ray Absorption Spectroscopy measurements and analyses performed on sediment samples will be included in future versions of the accompanying data package [12]. Depending on sample amounts remaining, additional analysis will be performed and appended to future versions of the data package.

1.5 Sediments

Surface sediments (i.e., top 5-10 cm of sediment) were collected into clear 2.5 oz PP jars and frozen at -20 ˚C upon arrival for archival purposes. One full plastic bag of sediment was also collected for gravimetric water content (GWC) and was stored at 4˚ C until analysis. Immediately upon arrival, subsamples from the sealed plastic bags were collected in minimal oxygen conditions and frozen for Fe XAFS analysis. X-ray Absorption Spectroscopy measurements and analyses performed on sediment samples will be included in future versions of the accompanying data package [12]. Depending on sample amounts remaining, additional analysis will be performed on archived samples and appended to future versions of the data package.

2. *Water Analyses*

2.1 Common water quality measurements (pH, ORP, conductivity, alkalinity)

Common water quality measurements (i.e., pH, ORP, conductivity, alkalinity) were performed on unfiltered water samples, within 24 hours of receiving. Samples were measured simultaneously for temperature, specific conductivity, oxidation-reduction potential, and alkalinity using a Mettler Toledo T7 auto-titrator equipped with an auto-sampler. Prior to starting each run and after every 5 samples, conductivity and pH sensors were checked with standards, and were recalibrated if outside the acceptable tolerance (+/- 1% for conductivity, and +/- 0.05 for pH). Conductivity was calibrated with a 50,000 µS/cm (+/- 1%) solution to cover the salinity range represented by samples (0 to ~35 PSU). pH was calibrated using a 3 point calibration curve (using calibration solutions of pH 4.01, 7.00, and 10.00). Alkalinity was determined by titration with 0.02 N HCl to an end-point of pH 4.00, following standard United States Geological Survey (USGS) procedures [13]. All water quality variables underwent quality control to flag values outside of sensor analytical ranges.

2.3 Dissolved Organic Carbon and Total Dissolved Nitrogen

Field-filtered samples were stored at 4 °C until analyzed for dissolved organic carbon (DOC) and total dissolved nitrogen (TDN). We performed DOC and TDN analyses simultaneously, within one week of sample collection on a Total Organic Carbon Analyzer (Shimadzu TOC-L). DOC was measured after in-line acidification with 1:12 hydrochloric acid, as non-purgeable organic carbon (NPOC) via catalytic combustion. TDN was measured by chemiluminescence. A combined carbon and nitrogen check standard was run every 10 samples. We visually inspected and confirmed the quality of calibration curves, check standards, and sample peak shapes prior to exporting data. Data underwent further quality control to flag values outside of the calibration curve and detection limit ranges.

2.4 Total Suspended Solids

Total suspended solids were measured on 1L grab samples, filtered within 24 hours of sample collection, following Environmental Protection Agency (EPA) method 160.2 [14] with slight modifications. Samples were filtered through pre-combusted and pre-weighed glass fiber filters (GFF, nominal pore size of 0.7μm). The filtrate was then filtered through 0.2 μm PES filters and stored at 4˚C until solid phase extraction procedures were performed. For TSS measurements, GF filters were dried in a 45 ˚C oven for 24-72 hours, until the filter mass was stable, and stored in a desiccator for 24-48 hours after drying until final weights were taken. Process blanks were filtered concurrently with sample filtering, and average blank signal was below detection. Total suspended solids were calculated gravimetrically as follows:

$$TSS\ mg/L = \frac{(Oven\ dry\ weight\ of\ sample\ and\ filter,\ in\ mg - oven\ dry\ weight\ of\ filter,\ in\ mg)\ x\ 1000\ mL/L}{volume\ filtered\ in\ mL}$$

The volume filtered in mL was determined via mass and correcting for density of water using temperature, pressure and salinity data obtained from the titrator (Section 2.1) with the package *gsw* [15] in R version 4.2.1. When the common water quality measurements samples were not collected, we gap filled these data by taking the average of all sites adjacent to the kit. Data underwent further quality control to flag values below the blank and above the reported method detection limit for the EPA method [14].

2.5 Colored Dissolved Organic Matter (CDOM)

UV absorbance scans and excitation-emission matrices (EEMs) were collected simultaneously with an Aqualog (Horiba Scientific) on filtered sub-samples which were stored at 4˚C until analysis. Absorbance was measured from 230 to 800 nm in 3 nm intervals, and blank corrected prior to exporting the data. EEMs were collected with the same wavelength constraints and further processed with drEEM toolbox v. 6.0 for Matlab [16] (https://www.openfluor.org). EEMs processing included blank correction, inner filter correction[17], and normalization to Raman Scatter units based on daily water Raman scans collected at an excitation of 350 nm.

2.6 High-Resolution Mass Spectrometry

Aliquots of SPE extracts described in Section 1.3 were normalized to a DOC concentration of 50 mg C/L prior to FTICR-MS analysis [18]. A 12 Tesla (12T) Bruker SolariX Fourier transform ion cyclotron resonance mass spectrometer (FTICR-MS) (Bruker, SolariX, Billerica, MA) located at the Environmental Molecular Sciences Laboratory (Richland, WA), was used to collect high-resolution mass spectra. Samples were injected directly into the instrument using a custom automated direct infusion cart that performed two offline blanks between each sample. The FTICR-MS was outfitted with a standard electrospray ionization (ESI) source, and data were acquired in negative mode with the needle voltage set to +4.0kV. Data were collected from 150 m/z – 1000 m/z at 8M. Three hundred scans were co-added for each sample and internally calibrated using OM homologous series separated by 14 Da (–CH2 groups). The mass measurement accuracy was typically within 1 ppm for singly charged ions across a broad m/z range (150 m/z - 1100 m/z). Bruker Data Analysis (version 5.0) was used to convert raw spectra to a list of m/z values by applying FTMS peak picker module with a signal-to-noise ratio (S/N) threshold set to 7 and absolute intensity threshold to the default value of 100. Chemical formulae were then assigned using Formularity [19], an in-house software, following the Compound Identification Algorithm [20–22]. Chemical formulae were assigned based on the following criteria: S/N >7, and mass measurement error < 0.5 ppm, taking into consideration the presence of C, H, O, N, S and P and excluding other elements [19]. Further processing of the data was done using the *fticrrr* R package [23], including: (a) removing peaks < 200 and > 800 m/z, (b) removing peaks associated with ^{13}C, and (c) blank correcting all spectra.

3. *Soil and Sediment Analyses*

3.1 Gravimetric Water Content

Gravimetric water content (GWC), as the dry moisture content, was determined following the protocol from [24], with slight modifications. Field moist soil (~5 g) was dried in the oven at 100 ˚C for 24 hours. Weight loss was then calculated using the following equation:

$$gwc\ (\%) = \frac{field\ moist\ weight - oven\ dry\ weight}{oven\ dry\ weight} \times 100$$

Dry weight basis is utilized herein to better indicate whether or not the soils were saturated or not.

3.2 Bulk Density

Bulk density was determined on intact cores (collected in HYPROP rings), calculated as:

$$bulk\ density\ (g/cm^3) = \frac{dry\ weight}{soil\ volume}$$

Samples in the HYPROP rings were field moist, so the following conversion was applied to estimate the dry weight in the above equation:

$$dry\ weight = \frac{wet\ weight}{(GWC/100) + 1}$$

3.3 Total Carbon and Nitrogen

Total carbon and nitrogen was determined on a percent weight basis by combustion and chromatographic separation using an ECS 8020 CHNS-O Elemental Analyzer (Orbit Technologies Pvt. Ltd.) equipped with a zero-blank electronic autosampler and thermal conductivity detector. Approximately 15 mg of freeze-dried, sieved and homogenized soil were weighed into tin capsules. Reaction and reduction columns were packed according to operation manual specifications for C/N mode. For sample analysis, furnace temperatures were set to 980 °C for the reaction column, 650 °C for the reduction column and 65 °C for the gas chromatograph. Carrier gas flow was held constant at ~110 ml/min. Standard reference materials were run prior to each sample set, immediately following the calibration curve. We confirmed software peak detection, peak identification and integrations prior to exporting data. Calibration curves and final sample weight percentages were calculated in R Version 4.2.1 with the package *EnvStats* [25].

3.4. Soil pH and conductivity

Soil pH and specific conductance were measured on freeze-dried and homogenized soils. Soil subsamples were shaken with deionized MilliQ water (1:10 weight:volume ratio) for 30 minutes and then analyzed using a Myron L 6PIIFCE pH and conductivity meter.

Data Records

Data are permanently deposited on the open access repository, Environmental Systems Science Data Infrastructure for a Virtual Ecosystem (ESS-DIVE), https://data.ess-dive.lbl.gov/datasets/doi:10.15485/1960313. Future data types will be added to the ESS-DIVE data package as they are completed and will be version-controlled in the **Change History** section of *README.pdf*.

The structure of the data package is as follows:

- ec1_README.pdf
- ec1_metadata_v1.zip
    - *ec1_dd.csv*: a file-level data descriptor file containing a list of every column present in the data files
    - *ec1_flmd.csv*: a file-level data descriptor file containing a list of every file name present in the data package
    - *ec1_sample_catalog.csv*: a file containing a list of all samples and their collection status or information about methodological inconsistencies
    - *ec1_metadata_kitlevel.csv*
    - *ec1_metadata_collectionlevel.csv*
    - *ec1_data_collectionlevel.csv*
    - *ec1_igsn_metadata.csv*
- ec1_soil_v1.zip
- ec1_sediment_v1.zip
- ec1_water_v1.zip

CSV file structure

- [Campaign]_[Sample Type]_[Analyte]_[QC level].csv
    - Ex. *ec1_soil_tctn_L2.csv*
    - Ex. *ec1_metadata_kitlevel_L2.csv*
- All .csv dataset files contain the following first three identifying columns:
    - *campaign*: coordinated sampling effort
    - *kit_id*: unique identifier for each collection of samples from a given site

    - *transect_location*: position along the coastal TAI transect (Figure 1)

DAT file structure

- [Kit_ID]_[Processing Step A]_[Processing Step ...Z].dat
    - Ex. *K004_DilCorr_IFE_RamNorm.dat*
    - Ex. *K013_DilCorr_Abs.dat*
- All .dat dataset files are organized by Kit_ID and in matrices.

Technical Validation

Technical validation steps were completed throughout the analysis process for each analyte (Figure 2). Quality assurance of sample integrity was maintained from sample kit receiving, assuring that the quality of each sample was not compromised, by monitoring temperature and container quality upon kit arrival. Instruments used to acquire EXCHANGE datasets were calibrated before each run and maintained using standard procedures for each instrument. Datasets were quality controlled following processing level designations, inspired by the Ameriflux and Fluxnet programs [26,27]. For Level 1 (L1) datasets, flags are provided but are not applied. L1 datasets were screened for a secondary review and calculating the limit of detection ranges. Normal procedures for data quality were implemented, such as blank correction, etc, as appropriate. Analytical replicates are averaged and outliers are also removed for L1 datasets. These datasets are archived on a Google Drive repository for additional data provenance and are available to EXCHANGE consortium. For Level 2 (L2) datasets, all flags are applied to the L1 datasets, flagged data points removed, and data are summarized based on categorical variables (e.g. Transect Location, Kit ID). Datasets available on ESS-DIVE include L2 data for concentration based datasets.

We adopted the use of ESS-DIVE's sample ID, file-level metadata, and CSV reporting formats [28–31] to increase the usability of this data package and generate findable, accessible, interoperable and reusable (FAIR) data for the coastal science community [31].

Usage Notes information related to github.

The COMPASS project and EXCHANGE share a strong commitment to open science and FAIR data practices, recognizing these principles as core values for effective collaborative research. EXCHANGE engages the wider research community by empowering its consortium members to define campaign goals and research priorities.

EXCHANGE openly shares data, methodology, and findings with the scientific community to ensure that the knowledge generated through the project is widely accessible. This dataset, along with all datasets from the COMPASS project, follows Creative Commons Attribution 4.0 licensing, making all data freely available to use and distribute via the ESS-DIVE repository. Additional analyses are being performed on these sample sets. The data repository (Pennington et al., 2023) will be updated periodically with such additional datasets, found at the same DOI, with version numbers of the data package indicating new datasets are available.

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Competing interests

The authors declare no conflict of interest.

Figures

Figure 1.

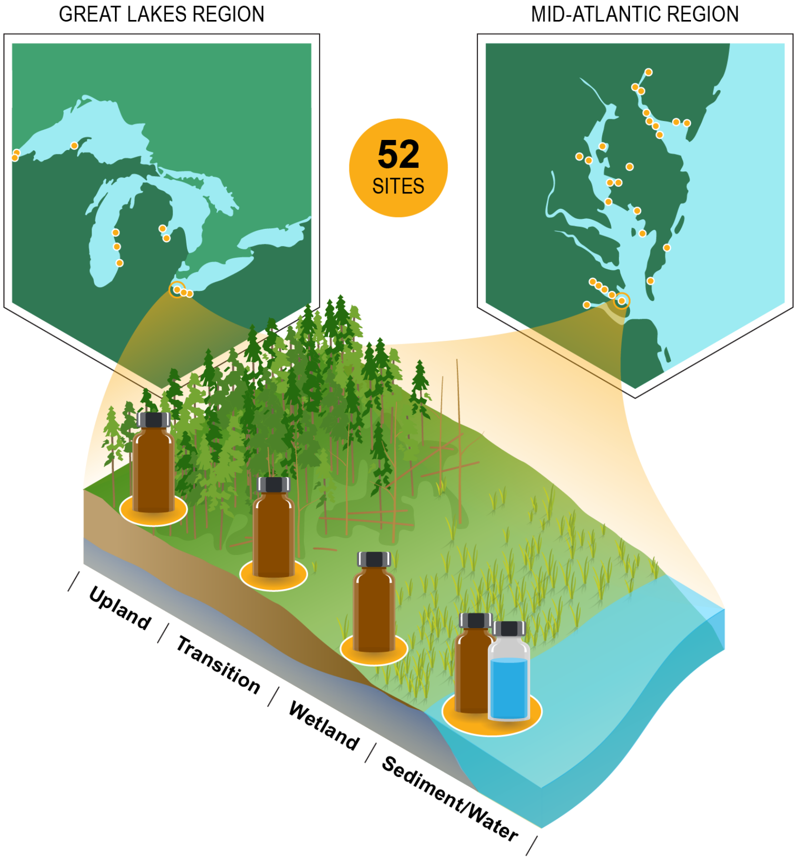

Figure 2.

Figure Legends

Figure 1. EXCHANGE campaign 1 sites were located in the Great Lakes and Mid-Atlantic Regions. 52 terrestrial-aquatic interfaces were sampled, from uplands to nearby waters (lake, estuary, stream, river, etc) for surface soils, sediments, and water samples.

Figure 2. Workflow of quality control procedures. Samples are received from the consortium, then processed at the Marine and Coastal Research Laboratory (PNNL–Sequim, WA) for analyses, which then were shared with the consortium and the public on ESS-DIVE.

Tables

Table 1. List of analyses included in the data package, including sample type and collection storage conditions.

Sample Type	Soil & Sediment				Water				
Collection	Intact cores *Soils only	Grab Samples			Unfiltered grab samples			Field-filtered grab samples	
Storage Method	4˚C	-20˚C	4˚C		4˚C			-20˚C	4˚C
Additional preparation for storage or analysis	–	Lyophilize, sieve	Subsample in anoxic environment	–	–	Filter in lab with GFF then 0.2µm PES, filtrate run through SPE, extract stored at -80˚ C	GFF filter dried at 45 ˚ C, then stored at room temperature in desiccator	–	–
Analyses performed	Bulk density Water retention curves Particle size/texture analysis	TC/TN pH Conductivity	Fe-EXAFS	Gravimetric water content	pH ORP Conductivity Alkalinity	FTICRMS	TSS	Major dissolved ions	DOC/TDN CDOM Water isotopes

Table 2. List of file names located in each .zip folder of the current version (V1) of the data package.

	Data Types
ReadMe	
Metadata	Metadata taken at the time of sample collection (Collection Level Metadata) Metadata taken for each kit (Kit Level Metadata) Data taken during each sample collection (Collection Level) Sample catalog Data dictionary of each column present in data package (DD) File-level metadata of each file present in data package (FLMD) IGSN sample metadata (IGSN Metadata)
Water Data	water quality (pH, ORP, alkalinity, conductivity) total suspended solids (TSS) dissolved organic carbon (DOC) total dissolved nitrogen (TDN) high-resolution mass spectrometry (FT-ICR-MS) colored dissolved organic matter absorbance and fluorescence (CDOM)
Sediment Data	gravimetric water content (GWC)
Soil Data	gravimetric water content (GWC) bulk density (BD) soil pH and conductivity (pH, Cond) total carbon (TC) total nitrogen (TN)

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