Thermogravimetric analysis (TGA) for precipitate from shipboard ocean alkalinity enhancement (OAE) experiments in the North Atlantic on R/V Atlantic Explorer cruise AE2320 in September 2023

Website: https://www.bco-dmo.org/dataset/963692
Data Type: Cruise Results, experimental
Version: 1
Version Date: 2025-06-11

Project
» OCE-PRF: Towards Quantifying Calcium Carbonate Sediment Dissolution During Marine Diagenesis (Marine CaCO3 and ocean chemistry)

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Abstract

To explore mineral formation due to alkalinity addition, we present results from shipboard experiments in which an aqueous solution of NaOH was added to unfiltered seawater collected from the surface ocean in the Sargasso Sea. Alkalinity addition ranged from 500 to 2000 micromoles per kilogram (µmol.kg-1) and the carbonate chemistry was monitored through time by measuring total alkalinity (TA) and dissolved inorganic carbon (DIC), which were used to calculate Ω. The amount of precipitate and its mineralogy were determined throughout the experiments. Mineral precipitation took place in all experiments over a timescale of hours to days. The dominant mineralogy of precipitate is aragonite with trace amounts of calcite and brucite. Aragonite crystallite size increases and its micro-strain decreases with time, consistent with Ostwald ripening. This dataset contains TGA analysis for precipitate from an experiment where TA was enhanced by 2000 µmol.kg-1.

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Coverage

Location: North Atlantic, Bermuda Atlantic Time Series

Spatial Extent: **Lat**:31.6667 **Lon**:-64.1667
Temporal Extent: 2023-09-05 - 2023-09-11

Methods & Sampling

Experiments involved the addition of NaOH solution prepared by weighing ACS grade NaOH in the lab prior to the cruise (R/V Atlantic Explorer AE2320) in a plastic Falcon tube that was capped and sealed with parafilm tape. During the cruise, DI water was added to make up stock NaOH solutions with a final concentration of 1 M. The NaOH solution was pipetted into the seawater-filled bags through the Luer-fitted stopcock. Because NaOH contributes only alkalinity but not DIC, seawater in the experiments was out of equilibrium with the atmosphere, which was intended to simulate conditions immediately following alkalinity addition to seawater during OAE deployments.

In total, 5 experiments were conducted. The first experiment (experiment A) was a control with no alkalinity addition. In the second, third, and fourth experiments (B, C, and D), alkalinity was enhanced by 500, 1000, and 2000 micromoles per kilogram (µmol/kg) respectively. The fifth experiment (E) represents a set of "sacrificial" time series experiments whereby 9 bags were prepared similar to other experiments and alkalinity was enhanced by 1000 µmol/kg in each one of them, but each bag was sequentially opened and filtered in order to evaluate the precipitate mineralogy through time. In experiment E, water samples for TA and DIC measurements were taken only at the end of the experiment. The experiments were run for approximately 5 days.

A TA Instruments Q600 simultaneous thermal analyzer was used for thermogravimetric analysis and differential scanning calorimetry of mineral precipitates from Experiment D. About 35 mg was heated from room temperature to 1100°C with a heating rate of 10°C per minute in a N2 atmosphere. The N2 flow rate was 50 mL.min-1. Routine measurements of standard materials supplied by the manufacturer indicate that the temperature is accurate to 1 degree Celsius (°C) and the weight change to 0.5 µg.

BCO-DMO Processing Description

- Imported original file "Hashim et al OAE resaerch data_TGA.xlsx" into the BCO-DMO system.
- Flagged "NA" as a missing data identifier (missing data are blank/empty in the final CSV file).
- Renamed fields to comply with BCO-DMO naming conventions.
- Saved the final file as "963692_v1_tga.csv"

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Data Files

File
963692_v1_tga.csv(Comma Separated Values (.csv), 899.95 KB) MD5:92090e9df3808041440509cb51e51f6c Primary data file for dataset ID 963692, version 1

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Related Publications

Hashim, M., Marx, L., Klein, F., Dean, C., Burdige, E., Hayden, M., McCorkle, D., & Subhas, A. (2025). Mineral Formation during Shipboard Ocean Alkalinity Enhancement Experiments in the North Atlantic. https://doi.org/10.5194/egusphere-2025-988
Results

Klein, F., Humphris, S. E., & Bach, W. (2020). Brucite formation and dissolution in oceanic serpentinite. Geochemical Perspectives Letters, 1–5. https://doi.org/10.7185/geochemlet.2035
Methods

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Parameters

Parameter	Description	Units
Time	The elapsed time since the start of the TGA experiment in minutes.	minutes
Temperature	The temperature of the sample during the analysis in degrees Celsius.	degrees Celsius
Weight_mg	The absolute mass of the sample at a given time/temperature in milligrams.	milligrams (mg)
Weight_pcnt	The relative mass of the sample, expressed as a percentage of the initial weight.	percent
first_derivative	The first derivative of the weight loss curve, indicating the rate of weight change, which helps identify decomposition events.	unitless
Heat_Flow	The thermal energy flow into or out of the sample in milliwatts	milliwatts (mW)
Temperature_Difference_C	The temperature difference between the sample and reference in degrees Celsius.	degrees Celsius
Temperature_Difference_uV	The temperature difference between the sample and reference expressed in microvolts	microvolts (uV)
Sample_Purge_Flow	The flow rate of the purge gas over the sample in milliliter per minute.	milliliters per minute (mL/min)

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Instruments

Dataset-specific Instrument Name	TA Instruments Q600
Generic Instrument Name	TA Instruments SDT Q600 simultaneous thermal analyzer
Dataset-specific Description	A TA Instruments Q600 simultaneous thermal analyzer was used for thermogravimetric analysis and differential scanning calorimetry of mineral precipitates.
Generic Instrument Description	The SDT Q600 is a combined thermogravimetric analyzer (TGA) and differential scanning calorimeter (DSC) that provides simultaneous measurement of weight change and true differential heat flow on the same sample from ambient to 1500 degrees C. It has a dual-beam design with automatic beam growth compensation and the ability to analyze 2 TGA samples simultaneously. DSC heat flow data is dynamically normalised using the instantaneous sample weight at any given temperature. The setup comprises a horizontal furnace encased in perforated stainless steel, a purge gas system with digital mass flow control, and a dual balance mechanism to enhance precision. The instrument has a DTA sensitivity of 0.001 degrees C, a calorimetric precision of +/- 2 %, and a heating rate of 0.1 to 25 degrees C per minute.

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Deployments

AE2320

Website	https://www.bco-dmo.org/deployment/963695
Platform	R/V Atlantic Explorer
Start Date	2023-09-04
End Date	2023-09-11
Description	See additional information from R2R: https://www.rvdata.us/search/cruise/AE2320

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Project Information

OCE-PRF: Towards Quantifying Calcium Carbonate Sediment Dissolution During Marine Diagenesis (Marine CaCO3 and ocean chemistry)

NSF Award Abstract:
OCE-PRF Towards Quantifying Calcium Carbonate Sediment Dissolution During Marine Diagenesis The goal of the project is to investigate dissolution of calcium carbonate (CaCO3) in sediments below the seafloor and determine its importance to the chemistry of seawater. This project uses sediment samples and chemical data collected from different parts of the ocean during the past five decades by scientific ocean drilling programs. Sediment dissolution of carbonate can lessen the impact of ocean acidification, the process that causes the pH of the ocean to decrease due to the uptake of carbon dioxide (CO2) from the atmosphere. Ocean acidification threatens the survival of marine organisms, such as oysters, clams, and coral reefs, which could alter marine food chains and food supply to humans. By improving understanding of carbonate dissolution in the ocean, results from this project will enable better predictions of the effects of ocean acidification on marine organisms. This will advance the progress of science and contribute to the knowledge that can inform public policy. In addition, understanding carbonate sediment dissolution serves other important purposes. For example, dissolution can create small spaces between sediments that may get filled with groundwater once sediments convert to rocks over millions of years. Thus, understanding the occurrence and spatial distribution of spaces

within rocks may help determine the volume and movement of groundwater in subsurface aquifers. This project provides support for a postdoctoral research fellow and research training opportunities for students through the Summer Student Fellowship and Woods Hole-wide Partnership Education Programs at the Woods Hole Oceanographic Institution.

Carbonate mineral dissolution is an integral part of the alkalinity and carbon cycles in the ocean and is expected to play an increasingly significant role in mediating changes in ocean chemistry as atmospheric CO2 continues to rise. The goal of this project is to provide thermodynamic constraints necessary for quantifying carbonate sediment dissolution in marine diagenetic environments. Specifically, the CaCO3 saturation state of pore fluids will be calculated in 365 globally distributed sites from previous scientific ocean drilling expeditions using a specially developed Pitzer ion activity model which is particularly useful for calculating activity coefficients in high ionic strength solutions such as those that characterize most diagenetic environments. These calculations will be substantiated with geochemical and textural analyses of sediment samples from four representative sites to identify the specific diagenetic processes (e.g., dissolution, precipitation, and recrystallization) and document the conditions responsible for their occurrence and prevalence. The immediate advantage of calculating the saturation state of pore fluids is that such data can be used to estimate carbonate sediment dissolution below the seafloor and quantify its contribution to the alkalinity and carbon cycles, which will lead to more accurate predictions of the consequences of ocean acidification. Another benefit of the global saturation state dataset is that it will improve our understanding of authigenic carbonate precipitation and its link to the carbon cycle over Earth history, which has been proposed as a significant sink for carbon. Furthermore, by complementing the thermodynamic calculations with textural and geochemical analyses, this project will parse out various diagenetic processes and identify the sedimentological and geochemical conditions responsible for their occurrence. Such knowledge is crucial for evaluating the impact of diagenesis on the carbonate-hosted paleoenvironmental proxies. Collectively, this project will pave the way towards a mechanistic understanding of carbonate diagenesis. This will provide important constraints on the oceanic alkalinity cycle, carbon burial rates, and geochemical proxies, which ultimately help us better understand the future of our ocean system in the context of climate change.

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-2205984

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