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A Report to the National Park Service on the Environmental Quality Assessment of Water and Sediment at Cape Lookout National Seashore

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INTRODUCTION

The National Centers for Coastal Ocean Science (NCCOS) is the focal point for NOAA's coastal ocean science efforts and helps NOAA meet its coastal stewardship and management responsibilities. It is responsible for executing NOAA's science mission: 1) to protect, restore and manage the use of coastal and ocean resources through an ecosystem approach to management; 2) to conserve and manage coastal and marine ecosystems and resources; 3) to recover marine and coastal species to health and productivity; and 4) share knowledge and information with others. NCCOS executes its responsibilities by providing coastal managers with scientific information to support management options for protecting environmental resources and public health, preserving valued habitats and improving community interactions with coastal ecosystems.

This project assessed water and sediment quality from 20 sites within Cape Lookout National Seashore. Oyster tissue from two sites were also sampled. Floodwaters and runoff from agricultural and industrial areas located upstream of the park are suspected of having chemical and bacterial contaminants, which could affect the environmental health of the area in addition to the health and safety of park visitors. In 2018, Hurricane Florence brought historic flooding to large regions of northern South Carolina and southern North Carolina. Like many other severe coastal storms, Hurricane Florence was suspected to cause significant damage to many ecological components of estuarine and coastal systems along coastal North and South Carolina. Inundation and freshwater influx into the coastal area may have had impacts on water quality and marine resources as result of salinity change, redistributed chemical pollution (e.g. pesticides, polycyclic aromatic hydrocarbons (PAHs), metals, nutrients), and runoff of bacterial pathogens from the impacted areas. Unfortunately, a lack of ecosystem assessment data pre-event inhibited quantification of coastal inundation impacts post-event and is a recognized gap in understanding potential storm impacts in federally protected resources such as Cape Lookout National Seashore. These types of extreme weather events may become more frequent and occur with increased intensity along the coastline of the Carolinas due to climate change. This project aims to provide critical baseline information regarding environmental quality within the National Seashore boundaries. This information will benefit environmental resource managers and coastal communities by assessing ecosystem vulnerabilities and providing a baseline for determining resource damages after an inundation event.

The area of study included 20 sites in Shackleford Banks, South Core Sound, and North Core Sound. The anticipated outcomes were the following:
1. Provide a critical baseline of sediment and water quality data from which to compare any future coastal inundation events to inform park management decisions regarding environmental and human protection.
2. Deliver a dataset for discrete sampling sites within the park boundaries of water quality to include nutrient and chlorophyll *a* levels and chemical contamination in sediments. Sample oyster tissue where available to provide background levels of biomarkers of contaminant exposure.
3. Develop a dataset that collects historical data from NOAA's Mussel Watch and other long-term monitoring programs in the region.

METHODS

Sediment, water, and oyster collection was accomplished during July - October 2021. Twenty sites were sampled as listed in Table 1 and mapped in Figure 1. The sites were accessed using NOAA Research Vessel R1807. This is an 18-foot, Class 1 vessel built by Privateer Boat Company and equipped with a Yamaha 90 hp outboard engine and a small davit to assist with sediment bottom grabs. The vessel was trailered to the appropriate boat landings for access to the sites. After navigating the vessel to each site, a 3-person crew (composed of a boat driver, boat and field support, and field support persons) spent approximately 1.50 hours to collect necessary water and sediment and record field data. The field data recorded at each site included latitude/longitude, surface water temperature (°C), salinity (parts per thousand [ppt]), dissolved oxygen (mg/L), pH, and Secchi depth.

At each site, approximately 500 mL of water was collected in amber glass bottles. These water samples were kept on ice for nutrient analysis and chlorophyll *a* analysis. For nutrient analysis, at least 250 mL of site water was filtered through a 47 mm GF/F filter membrane. The filtrate was then placed in a polypropylene bottle and frozen until analysis. Nutrient analysis for ammonium, nitrate plus nitrite, and phosphate was performed by Nutrient Analytical Services Laboratory (University of Maryland Center for Environmental Science, Chesapeake Biological Laboratory, 146 Williams Street, P.O. Box 38, Solomons, MD 20688) using methods as posted on their website (https://www.umces.edu/nasl/methods). For chlorophyll *a* analysis, at least 10 mL of site water was filtered through a 25 mm GF/F filter membrane. If no color was present on the filter, then additional 10 mL aliquots of site water was filtered until coloration was present. The filter was placed face up in a scintillation vial with 1 mL of saturated magnesium carbonate solution, and frozen. For analysis, 9 mL of acetone was added to each vial, shaken, then refrigerated. After 24 h, the vials were shaken and refrigerated overnight again. The chlorophyll *a* extracts were then read on a Sequoia-Turner Model 450 fluorometer. Readings were calculated then converted to µg/L after taking into account the door factor, gain correction, and volume filtered (Glover and Morris, 1979).

At each site, approximately 750 mL of sediment was collected using a Young-modified Van Veen sampler as based on Apeti et al. (2018). At least three of these bottom samples were collected at each site. The surficial sediments (upper 2 cm) of each grab was homogenized on-site in a disposable polypropylene liner placed inside a five-gallon plastic bucket. Sediment was then placed in pre-cleaned containers for analysis of total organic carbon (TOC), contaminants, and sediment toxicity. After enough sediment had been collected, the grab and all utensils were rinsed with the site water then cleaned with a iso-propyl saturated disposable cloth. A new liner was also placed in the five-gallon bucket ready for the next site.

All sediment samples were kept on ice while in the field and then stored either at 4°C (toxicity) or frozen (contaminants, TOC) until analyzed. Contaminants measured in sediment included 22 metals, 80 pharmaceuticals and personal care products, 17 human hormones, 50 polycyclic aromatic hydrocarbons (PAHs), 4 alkylphenols, and 25 pesticides. These contaminants were analyzed by the NOAA/NOS National Centers for Coastal Ocean Science Charleston Laboratory using procedures similar to those described by Kucklick et al. (1997), Long et al. (1997), Balthis et al. (2012), and Chen et al. (2012). Also measured were 84 contaminants of emerging concern (CECs) which were analyzed by the NOAA/NOS National Centers for Coastal Ocean Science Charleston Laboratory using procedures similar to those described by Apeti et al (2018). In this study, measurement of PFAS compounds (per- and polyfluoroalkyl substances) in sediment and tissue samples was conducted by TDI Brooks/B&B Laboratories (14391B South Dowling Road, College Station, TX 77845). These analytical methods are proprietary and confidential. Sediment toxicity was assessed by the Microtox® solid-phase bioassay, which uses a photoluminescent bacterium (*Vibrio fischeri*) and protocols described by the Microbics Corporation (1992) and Ringwood et al. (1997).

Oysters were collected from two sites, CL2021 - 007 and 019. Oysters were not present at the other sites sampled. Oyster tissue, hepatopancreas and gill, were dissected and flash frozen to be analyzed for cellular stress using two bioassays, lipid peroxidation and glutathione, as based on methods from Ringwood et al. (2003), Hoguet and Key (2007), and Aquilina-Beck et al. (2020). Oyster tissue was also analyzed for contaminants by TDI Brooks/B&B Laboratories using proprietary and confidential methods.

RESULTS and DISCUSSION

Water

The 20 sites that were sampled in Cape Lookout National seashore are listed in Table 1 along with corresponding depth, Secchi depth (as a measure of turbidity), temperature, and salinity. The current water quality standards for surface waters in North Carolina as developed by North Carolina Department of Environmental Quality, Division of Water Resources can be viewed here (https://deq.nc.gov/ncstdssurfacetrirevfinalrules2018) in rules 15A NCAC 02B .0100 through .0300. Dissolved oxygen in tidal salt water in NC should not be less than 5.0 mg/L. pH should be between 6.8 and 8.5. All sites sampled were within the ranges for dissolved oxygen and pH. Dissolved oxygen ranged from 5.54 - 7.09 mg/L while pH at the 20 sites ranged from 7.8 - 8.3. Observations with the Secchi disk indicated that turbidity was low at these sites.

Nutrients and chlorophyll *a* levels measured in water at the sampled sites are listed in Table 2. According to NC DEQ guidelines (https://deq.nc.gov/ncstdssurfacetrirevfinalrules2018), chlorophyll *a* levels should not exceed 40 µg/L for all waters not designated as trout waters. Nitrite/Nitrate levels for NC should not exceed 10 mg/L for any surface waters. Ammonia standards follow that of US EPA document number 440/5-88-004; NTIS number PB89-169825. Phosphate standards could not be found for NC. EPA phosphate standards indicate levels should be no more than 0.1 mg/L for streams that do not empty into reservoirs; no more than 0.05 mg/L for streams discharging into reservoirs; and no more than 0.024 mg/L for reservoirs (UCD ExtoxNet FAQ Team, U.S. Environmental Protection Agency, Water Research Center). All nutrients measured at the Cape Lookout sites were below NC and EPA levels of concern (Table 2). The chlorophyll *a* levels were also below levels of concern (Table 2).

Sediment

Sediment from all 20 sites was analyzed for a variety of contaminants, most of which were not detected. All pharmaceutical and personal care products (PPCPs) and human hormones for which the sediment was analyzed were below the method detection limits (Tables 3 and 4). The PPCPs listed include prescription and over-the-counter medications, synthetic fragrances, disinfectants, antimicrobial compounds, disinfectants, and insect repellants. A primary route for PPCPs and hormones to enter the environment would be through wastewater discharge or direct disposal (Apeti et al., 2018). More information on the chemical group and general use of these PPCPs and hormones may be found in Apeti et al. (2018). The lack of detection of these compounds at Cape Lookout may indicate that, at the time of sampling, any wastewater discharge and/or improper disposal was not a concern.

Of the 25 pesticides that were measured (Table 5A), only three pesticides were detected (4,4'-DDD, heptachlor epoxide, cis-chlordane) but only one each at five different sites (CL2021-003, CL2021-004, CL2021-007, CL2021-018, CL2021-019) (Table 5B), all at low ng/g levels. The three detected are metabolites of organochlorine insecticides that were used in termite and mosquito control in addition to being used on agricultural crops. DDT was banned in 1972 and chlordane and heptachlor were banned in 1988, (https://wwwn.cdc.gov/TSP/substances/ToxSubstance.aspx?toxid=20; https://wwwn.cdc.gov/TSP/ToxFAQs/ToxFAQsDetails.aspx?faqid=354&toxid=62#:~:text=Chlordane%20is%20a%20man%2Dmade,lawns%2C%20gardens%2C%20and%20homes;

https://archive.epa.gov/epawaste/hazard/wastemin/web/pdf/hepchlep.pdf). Their continued presence years after severely limiting and ultimately banning their use is testament to their persistence. The level of pesticides found in the sediment can be compared against a series of sediment quality guidelines developed by Long et al. (1995) known as Effects Range Low (ERL) and Effects Range Medium (ERM). The ERL value represents a chemical level in which effects to sediment dwelling estuarine organisms would be rarely observed (<10% of the time). The ERM value represents a chemical level in which adverse effects would frequently occur (~50% of the observations). Between the two values, represents a range in which effects would occasionally occur (Long et al., 1995). The ERL and ERM values that have been determined so far are seen in Table 9. The levels of 4,4'-DDD, heptachlor epoxide, and cis-chlordane found at these sites indicate that effects on sediment dwelling organisms would not likely be observed based on these concentrations.

Sediment was analyzed for four alkylphenols (Table 6A), but only one was detected (4-n-octylphenol) and at four of the 20 sites (CL2021-013, CL2021-015, CL2021-017, CL2021-019; Table 6B). Alkylphenols are chemicals used as detergents and surfactants that tend to be persistent in the environment. The source of these compounds in the environment would be wastewater discharge (Apeti et al., 2018). The one alkylphenol detected was at ng/g levels.

At the 20 sites, total PAH50s were detected at 18 sites (Table 7A; see Table 7B for list of total PAH50 compounds). At sites CL2021-003 and CL2021-005, total PAH50s were not detected. These 50 PAHs, including both parent and alkylated PAHs, are known as "total PAH50" and represent a suite of target analytes in environmental petroleum samples (Boehm, 2006). PAHs are generated from the combustion of fossil fuels and can be persistent in sediments such as marsh substrates especially when the substrates have a high organic carbon content (Vo et al., 2004). The amount of total PAHs found at site CL2021-019, 295.1 ng/g, was higher than all the other sites. Sites CL2021-002, CL2021-007, CL2021-010, CL2021-020 had the next highest measured total PAH levels. However, none of the detectable levels exceeded the ERL values for total PAHs indicating that effects on sediment dwelling organisms would not likely be observed based on these PAH concentrations.

Metals in sediments were detected in all 20 sites (Table 8). Of the 22 metals measured, 13 were found in all 20 sites. Three of the metals (silver, cadmium, antimony) were not found at any site. Metals were detected at the highest amounts at sites CL2021-019 and CL2021-020. At both of these sites, 19 metals were detected. At these two sites, aluminum was found at levels from 15,804 to 18,405 µg/g which was seven times higher than the next highest site (CL2021-002). Iron was found at CL2021-019 and CL2021-020 at levels from 13,620.8 to 14,701 µg/g which was four times higher than the next highest site (CL2021-002 again). Nine of the metals for which Cape Lookout sediments were analyzed have ERL and ERM values (Table 9). However, none of the detectable levels exceeded the ERL values for those metals indicating that effects on sediment dwelling organisms would not be observed based on these metal concentrations.

PFAS levels in sediments at the 20 sites will be forthcoming from TDI Brooks/B&B Laboratories (Table 10). PFAS are a group of fabricated chemicals that have been used in a variety of industries since the 1940s including food packaging, commercial household products, and electronics manufacturing. These chemicals are very persistent in the environment and have been found in fish, animals, and humans where they have the ability to build up and persist over time.

Microtox analysis is a measure of sediment toxicity using a luminescent bacterium (*V. fischeri*). The values reported in Table 11 are the effective concentration of a sediment slurry that caused a 50% reduction in light emission from the bacteria over a set time period. The practical use of these EC50 values depends upon the percentage of silt and clay in the sediment samples. This information will be forthcoming from TDI Brooks/B&B Laboratories.

Oyster Tissue

Oysters collected from sites CL2021-007 and CL2021-019 were analyzed for two biomarkers commonly used to assess cellular stress. Lipid peroxidation (LPx), measured as malondialdehyde (MDA), ranged from a mean of 413 nmol/g MDA to 432 nmol/g MDA (Figure 2). According to Ringwood et al. (2002), a normal level of LPx in oysters would be <150 nmol/g. With the levels seen here (Figure 2), the oysters at both sites were showing signs of cellular membrane damage. Glutathione (GSH) levels (Figure 3) measured in the oysters from these two sites fell within the normal range of 800–1600 nmol/g (Ringwood et al. 2002), indicating that this phase II enzyme system, used to detoxify contaminants and provide antioxidant defense, was functioning normally.

Chemical analysis of oyster tissue collected from sites CL2021-007 and CL2021-019 will be forthcoming (Table 12).

CONCLUSIONS

Results from the analysis of water collected from 20 sites in Cape Lookout National Seashore found that the nutrient levels measured were below levels of concern according to North Carolina and EPA standards. The water quality parameters measured at the time of sampling were also within normal ranges. Chlorophyll *a* analysis indicated that there were no samples that exceeded NC guidelines indicating that eutrophication is not a concern at the sites sampled. Oyster tissue biomarker analysis indicated some cellular stress but the stress cannot be attributed to specific environmental conditions. Chemical analysis of the oyster tissue may better clarify any chemical to biomarker links once available from the contract lab TDI Brooks/B&B Laboratories.

Results from the analysis of sediment from these 20 sites found at least one class of contaminants at all 20 sites. Two sites, CL2021-019 and -020, contained the highest levels and number of metals and the highest levels of total PAH50s. The field crew noted that these two sites are close to the ferry path which runs from Ocracoke to Cedar Island, as well as being close to Ocracoke Inlet through which boats may pass. CL2021-019 was sampled near a dock used to service a residence in the abandoned town of Portsmouth. Cl2021-020 was sampled in a channel leading to an abandoned Coast Guard station. While these sites found contaminants in the sediment that are often attributed to combustion of oil, the contaminant levels measured did not exceed sediment guidelines where an effect would be expected. Further chemical analysis of sediment samples is forthcoming by the contract lab TDI Brooks/B&B Laboratories.

The data presented in this report gives much needed background levels of nutrients and contaminants along a transect in Cape Lookout National Seashore. Environmental monitoring of this region will help to determine effects of floodwaters from natural weather events, mainland runoff from upstream agricultural and industrial areas, and effects of recreational and commercial boating within park boundaries. These results will provide necessary data for future management and comparison sampling. For previous NOAA studies on the state of North Carolina's coastal zone, please see the reference list below.

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Table 1. List of sites sampled with water quality parameters in Cape Lookout National Seashore.

Site Name	Latitude (DD)	Longitude (DD)	Matrix	Date	Time	Depth (m)	Secchi Depth (m)	Temp (°C)	Salinity (ppt)
CL2021-001	34.68713	-76.64529	Sediment/Water	7/27/2021	14:24	1.0	1.0	28.9	35.5
CL2021-002	34.67136	-76.59474	Sediment/Water	7/27/2021	13.46	1.6	1.4	29.3	35.31
CL2021-003	34.63638	-76.52646	Sediment/Water	7/27/2021	10:53	1.4	1.4	28.2	35.54
CL2021-004	34.64207	-76.52747	Sediment/Water	7/27/2021	10:22	1.4	1.4	28.1	35.49
CL2021-005	34.64528	-76.52970	Sediment/Water	7/27/2021	09:45	1.2	1.2	27.9	35.45
CL2021-006	34.61567	-76.52983	Sediment/Water	7/27/2021	12:35	1.3	1.3	28.7	35.44
CL2021-007	34.61215	-76.53754	Sediment/Water/Oysters	7/27/2021	11:57	2.3	2.3	28.5	35.39
CL2021-008	34.61459	-76.54798	Sediment/Water	7/27/2021	11:25	1.3	1.3	28.4	35.54
CL2021-009	34.68687	-76.50105	Sediment/Water	7/28/2021	14:23	0.6	0.6	29.2	35.51
CL2021-010	34.76914	-76.42194	Sediment/Water	7/28/2021	12:30	1.7	1.7	29.6	36.14
CL2021-011	34.81447	-76.38739	Sediment/Water	7/30/2021	10:45	1.4	1.4	27.4	35.9
CL2021-012	34.83038	-76.37008	Sediment/Water	7/30/2021	10:00	0.8	0.8	27.6	35.98
CL2021-013	34.87738	-76.30556	Sediment/Water	7/29/2021	14:03	1.2	1.2	29.2	34.77
CL2021-014	34.90209	-76.26803	Sediment/Water	7/29/2021	13:22	1.1	1.1	29.2	35.44
CL2021-015	34.94987	-76.21571	Sediment/Water	7/29/2021	12:43	0.8	0.8	29.6	22.35
CL2021-016	34.9959	-76.17521	Sediment/Water	7/29/2021	10:25	1.3	1.3	27.8	24.26
CL2021-017	35.04943	-76.10895	Sediment/Water	7/29/2021	09:24	1.3	1.3	27.9	21.06
CL2021-018	35.06552	-76.08483	Sediment/Water	7/29/2021	08:41	0.8	0.8	27.1	22.68
CL2021-019	35.07090	-76.06851	Sediment/Water/Oysters	10/12/2021	09:45	2.8	NR	21.1	27.0
CL2021-020	35.07086	-76.05644	Sediment/Water	10/12/2021	10:35	2.0	NR	22.1	26.0

Temp = temperature; NR = not recorded

Table 2. Measured nutrient levels and chlorophyll *a* levels in surface water at the sampled sites. Nutrient levels for sites 019 and 020 will be forthcoming. MDL = method detection limit

Site Name	Ammonia* (NH4) [mg N/L]	Phosphate** (PO4) [mg P/L]	Nitrite plus Nitrate*** (NO3+NO2) [mg N/L]	Chlorophyll *a* [µg/L]
CL2021-001	<MDL	0.0035	0.0016	2.28
CL2021-002	<MDL	<MDL	<MDL	2.34
CL2021-003	<MDL	<MDL	<MDL	0.407
CL2021-004	<MDL	<MDL	0.0038	1.29
CL2021-005	<MDL	0.0035	0.0034	1.11
CL2021-006	<MDL	<MDL	<MDL	21.90
CL2021-007	<MDL	0.0039	<MDL	1.55
CL2021-008	<MDL	<MDL	<MDL	0.78
CL2021-009	<MDL	<MDL	<MDL	2.42
CL2021-010	<MDL	<MDL	<MDL	2.05
CL2021-011	<MDL	<MDL	0.0047	3.55
CL2021-012	<MDL	<MDL	<MDL	6.20
CL2021-013	<MDL	<MDL	<MDL	3.36
CL2021-014	<MDL	<MDL	0.0021	6.22
CL2021-015	<MDL	0.0087	<MDL	9.58
CL2021-016	<MDL	<MDL	<MDL	9.07
CL2021-017	<MDL	<MDL	<MDL	9.67
CL2021-018	<MDL	<MDL	<MDL	1.07
CL2021-019				11.00
CL2021-020				19.20

*MDL = 0.009 mg N/L

**MDL = 0.0035 mg P/L

***MDL = 0.0015 mg N/L

Table 3. List of pharmaceutical and personal care products for which Cape Lookout sediments were analyzed. All products were below the method detection limit (ng/g levels).

acetaminophen	2-hydroxy ibuprofen	albuterol	10-hydroxy-amitriptyline
caffeine	bisphenol-a	atenolol	alprazolam
carbamazepine	furosemide	atorvastatin	amitriptyline
clarithromycin	gemfibrozil	cimetidine	amlodipine
cloxacillin	glipizide	clonidine	benzoylecgonine
dehydronifedipine	glyburide	cotinine	benztropine
digoxigenin	hydrochlorothiazide	enalapril	betamethasone
diphenhydramine	ibuprofen	hydrocodone	cocaine
fluoxetine	naproxen	oxycodone	deet
norfloxacin	triclocarban	triamterene	diazepam
ofloxacin	triclosan	busulfan	fluocinonide
oxacillin	warfarin	citalopram	fluticasone propionate
paraxanthine		clotrimazole	hydrocortisone
penicillin g		etoposide	meprobamate
penicillin v		venlafaxine	methylprednisolone
sulfachloropyridazine			metprolol
sulfadiazine			n-desmethyldiltiazem
sulfadimethoxine			norfluoxetine
sulfamerazine			norverapamil
sulfamethazine			paroxetine
sulfamethizole			prednisolone
sulfamethoxazole			prednisone
sulfanilamide			propoxyphene
sulfathiazole			propranolol
thiabendazole			sertraline
trimethoprim			verapamil
tylosin			

Table 4. List of human hormones for which Cape Lookout sediments were analyzed. All products were below the method detection limit (ng/g levels).

17a-dihydroequilin	allyl trenbolone
17a-estradiol	androstenedione
17a-ethynyl estradiol	desogestrel
17B-estradiol	mestranol
diethylstilbestrol	norethindrone
equilenin	norgestrel
equilin	progesterone
estriol	testosterone
estrone	

Table 5A. List of pesticides for which Cape Lookout sediments were analyzed. All fell below method detection limits (ng/g levels) except for those in Table 5B.

aldrin	2,4'-DDD
alpha-hch	2,4'-DDE
beta-hch	2,4'-DDT
chlorpyrifos	4,4'-DDD
cis-chlordane (alpha-chlordane)	4,4'-DDE
cis-nonachlor	4,4'-DDT
dieldrin	
endosulfan i	
endosulfan ii	
endosulfan sulfate	
endrin	
gamma-chlordane	
gamma-hch (g-bhc, lindane)	
heptachlor	
heptachlor epoxide	
hexachlorobenzene	
mirex	
oxychlordane	
trans-nonachlor	

Table 5B. Cape Lookout sites for which pesticides were detected in sediments above the method detection limit.

Site name	Pesticide	Amount (ng/g dry mass)
CL2021-003	4,4'-DDD	0.041
CL2021-004	4,4'-DDD	0.064
CL2021-007	4,4'-DDD	0.053
CL2021-018	heptachlor epoxide	0.030
CL2021-019	cis-chlordane	0.018

Table 6A. List of alkylphenols for which Cape Lookout sediments were analyzed. All fell below method detection limits (ng/g levels) except for those in Table 6B.

4-n-octylphenol
4-nonylphenol
NP1EO
NP2EO

Table 6B. Cape Lookout sites for which alkylphenols were detected in sediments above method detection limits.

Site Name	4-n-octylphenol (ng/g)
CL2021-013	2.71
CL2021-015	4.499
CL2021-017	1.36
CL2021-019	0.971

Table 7A. Analysis of total PAH50 in Cape Lookout sediments. MDL = method detection limit. See Table 7B below for list of total PAH50.

Site Name	total PAH50 (ng/g dry mass)
CL2021-001	0.103231
CL2021-002	37.049357
CL2021-003	<MDL
CL2021-004	3.641229
CL2021-005	<MDL
CL2021-006	0.977966
CL2021-007	32.959062
CL2021-008	3.761089
CL2021-009	4.273893
CL2021-010	26.882329
CL2021-011	1.131759
CL2021-012	0.179918
CL2021-013	0.784357
CL2021-014	1.268647
CL2021-015	0.295596
CL2021-016	3.481435
CL2021-017	1.480974
CL2021-018	3.020806
CL2021-019	295.139376
CL2021-020	139.074012

Table 7B. The parent and alkylated PAH analytes measured in total PAH50 as defined by Boehm (2006).

PAH50 Analytes	
Parent PAH	**Alkylated PAH**
naphthalene	C1-naphthalenes
biphenyl	C2-naphthalenes
Acenaphthene	C3-naphthalenes
acenaphthylene	C4-naphthalenes
fluorene	C1-fluorenes
dibenzofuran	C2-fluorenes
dibenzothiophene	C3-fluorenes
phenanthrene	C1-dibenzothiophenes
anthracene	C2-dibenzothiophenes
fluoranthene	C3-dibenzothiophenes
pyrene	C4-dibenzothiophenes
benz(a)anthracene	C1-phenanthrenes/anthracenes
benzo(b)naphtho(2,1-d)thiophene	C2-phenanthrenes/anthracenes
chrysene+triphenylene	C3-phenanthrenes/anthracenes
benzo(a)fluoranthene	C4-phenanthrenes/anthracenes
benzo(b)fluoranthene	C1-fluoranthenes/pyrenes
benzo(j)fluoranthene	C2-fluoranthenes/pyrenes
benzo(k)fluoranthene	C3-fluoranthenes/pyrenes
benzo(a)pyrene	C4-fluoranthenes/pyrenes
benzo(e)pyrene	C1-chrysenes/benzanthracenes
dibenzo(a,h)anthracene	C2-chrysenes/benzanthracenes
indeno(1,2,3-c,d)pyrene	C3-chrysenes/benzanthracenes
benzo(g,h,i)perylene	C4-chrysenes/benzanthracenes
	C1-naphthobenzothiophenes
	C2-naphthobenzothiophenes
	C3-naphthobenzothiophenes
	C4-naphthobenzothiophenes

Table 8. Analysis of metals in Cape Lookout sediments. All values are µg/g dry mass. MDL = method detection limit.
MDLs: Ag ≤ 0.228; As ≤0.258; Be ≤0.073; Cd ≤0.198; Cu ≤0.721; Sb ≤1.21; Sn ≤0.24; Ti ≤0.115; U ≤0.296

Site Name	Al	As	Ba	Be	Co	Cr	Cu	Fe	Hg	Li	Mn	Ni	Pb	Se	Sn	Tl	U	V	Zn
CL2021-001	693.5	1.0	4.2	0.1	0.6	4.8	<MDL	1512.6	0.002	2.5	11.5	1.0	1.5	0.3	<MDL	<MDL	<MDL	2.7	4.3
CL2021-002	2573.6	2.6	7.3	0.2	1.3	8.5	1.8	3647.9	0.007	6.7	29.8	2.8	2.8	0.7	<MDL	<MDL	0.3	7.6	10.3
CL2021-003	510.0	0.6	2.9	<MDL	0.4	3.7	0.7	937.5	0.002	2.2	10.6	0.8	1.9	0.5	<MDL	<MDL	<MDL	2.4	3.3
CL2021-004	1533.2	1.1	6.2	0.1	0.8	5.5	1.1	2086.3	0.004	4.2	18.3	1.7	2.0	0.6	<MDL	<MDL	<MDL	4.6	6.1
CL2021-005	298.8	0.8	2.1	<MDL	0.3	2.2	<MDL	536.1	0.002	1.5	4.5	0.8	0.9	0.6	<MDL	<MDL	<MDL	1.6	2.4
CL2021-006	529.3	<MDL	3.8	<MDL	0.5	3.5	<MDL	936.7	0.002	2.2	11.8	1.5	2.3	0.5	<MDL	<MDL	<MDL	2.6	3.7
CL2021-007	738.7	1.0	4.6	<MDL	0.6	3.7	1.2	1496.4	0.007	2.7	11.2	3.2	1.6	0.7	<MDL	<MDL	<MDL	3.4	4.3
CL2021-008	432.3	1.3	3.0	<MDL	0.5	2.7	<MDL	1226.1	0.002	1.9	10.4	2.6	0.8	0.7	<MDL	<MDL	<MDL	2.9	2.6
CL2021-009	672.4	0.6	4.9	<MDL	0.5	3.6	<MDL	927.0	0.003	2.5	7.8	0.7	1.0	0.3	<MDL	<MDL	<MDL	1.9	3.5
CL2021-010	1282.8	0.6	6.4	0.1	0.8	4.7	0.8	1874.9	0.006	3.9	16.2	1.3	1.6	0.3	<MDL	<MDL	<MDL	3.5	5.8
CL2021-011	866.3	0.5	5.2	0.1	0.7	4.3	2.4	1265.2	0.003	3.6	14.2	1.0	2.2	0.5	<MDL	<MDL	<MDL	2.8	5.2
CL2021-012	742.8	0.4	5.9	<MDL	0.6	4.4	<MDL	1069.9	0.003	2.9	11.7	0.8	1.7	0.3	<MDL	<MDL	<MDL	2.4	5.6
CL2021-013	603.5	0.8	4.4	0.1	0.6	3.7	<MDL	840.4	0.005	2.6	6.9	0.9	0.9	0.4	<MDL	<MDL	<MDL	1.8	3.4
CL2021-014	832.2	0.3	6.4	0.1	0.6	4.3	<MDL	1164.2	0.004	3.1	10.8	0.8	1.7	0.2	<MDL	<MDL	0.4	2.7	4.5
CL2021-015	637.5	<MDL	4.9	<MDL	0.5	4.1	<MDL	918.4	0.003	2.7	11.0	0.8	1.8	0.6	<MDL	<MDL	<MDL	2.6	3.9
CL2021-016	911.0	0.5	6.0	<MDL	0.6	3.3	0.9	1176.3	0.005	3.1	9.7	0.8	1.2	0.2	<MDL	<MDL	<MDL	2.1	7.3
CL2021-017	538.1	0.4	4.3	<MDL	0.4	2.5	<MDL	671.6	0.003	2.4	5.2	0.6	0.7	0.2	<MDL	<MDL	<MDL	1.1	2.8
CL2021-018	1606.4	0.5	4.1	0.1	0.7	4.8	<MDL	1608.1	0.005	4.0	18.9	1.0	1.5	0.3	<MDL	<MDL	<MDL	2.8	5.1
CL2021-019	15804.5	5.1	38.8	0.6	4.6	26.5	6.0	13620.8	0.016	26.6	141.0	9.5	7.1	1.3	0.7	0.2	0.8	26.3	34.7
CL2021-020	18405.0	5.0	42.7	0.7	4.9	29.2	6.7	14701.0	0.019	29.7	123.6	11.1	8.6	1.3	0.8	0.2	1.2	30.8	39.8

Table 9. Effects Range Low (ERL) and Effects Range Medium (ERM) values for metals and organic compounds commonly found in sediments. Based on Long et al. (1995).

	ERL Guideline	ERM Guideline
Metals (µg/g)		
Arsenic	8.2	70
Cadmium	1.2	9.6
Chromium	81	370
Copper	34	270
Lead	46.7	218
Mercury	0.15	0.71
Nickel	20.9	46.1
Silver	1	3.7
Zinc	150	410
Organic compounds (ng/g)		
Acenaphthene	16	500
Acenaphthylene	44	640
Anthracene	85.3	1,100
Benzo[*a*]anthracene	261	1,600
Benzo[*a*]pyrene	430	1,600
Chrysene	384	2,800
Dibenz[*a,h*]anthracene	63.4	260
Fluoranthene	600	5,100
Fluorene	19	540
2-Methylnaphthalene	70	670
Naphthalene	160	2,100
Phenanthrene	240	1,500
Pyrene	665	2,600
Total PAHs	4020	44,792
Chlordane	0.5	6
4,4'-DDD	2	20
4,4'-DDE	2.2	27
Total DDTs	1.58	46.1
Total PCBs	22.7	180

Table 10. PFAS concentrations in sediment from 20 Cape Lookout sites.
To be included once data is available.

Table 11. Microtox effective concentration (EC50) of sediment that causes a 50% reduction in light emission of *Vibrio fischeri*. EC50 corrected for dry weight of sediment. % silt/clay results will be included once data is available.

Site Name	EC50 (%)	% silt/clay
CL2021-001	>15.4402	
CL2021-002	1.3795	
CL2021-003	>15.3935	
CL2021-004	>16.0873	
CL2021-005	>16.3231	
CL2021-006	3.5107	
CL2021-007	3.7395	
CL2021-008	2.2976	
CL2021-009	>15.4160	
CL2021-010	7.6729	
CL2021-011	12.2821	
CL2021-012	>16.0180	
CL2021-013	>14.2205	
CL2021-014	10.6987	
CL2021-015	>15.7058	
CL2021-016	>15.0999	
CL2021-017	>15.0969	
CL2021-018	>14.9733	
CL2021-019	0.2948	
CL2021-020	0.2077	

Table 12. Contaminants found in oyster tissue from two sites from Cape Lookout National Seashore. To be included once data is available.

Figure 1. Sites sampled in Cape Lookout National Seashore.

Figure 2. Results of lipid peroxidation, expressed as mean ± S.E. malondialdehyde (MDA), measured in oyster hepatopancreas tissue (n=3) collected from two sites at Cape Lookout National Seashore.

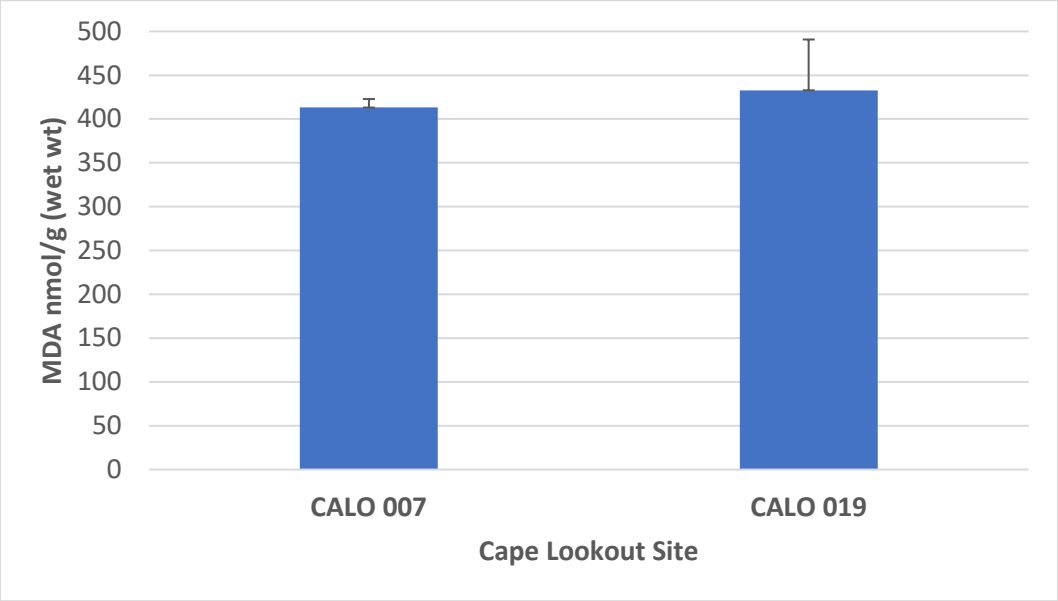

Figure 3. Results of glutathione (mean ± S.E. GSH) measured in oyster hepatopancreas tissue (n=3) collected from two sites at Cape Lookout National Seashore.