Interim Report – Project ER-201131

SHORT-TERM PERFORMANCE OF AN ACTIVATED CARBON AMENDMENT TO REDUCE PCB BIOAVAILABILITY AT AN ACTIVE NAVAL SHIPYARD

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Contents

Abbreviations and Acronyms

AC – activated carbon
BC – black carbon
BNC – Bremerton Naval Complex
CAD – confined aquatic disposal
CERCLA – Comprehensive Environmental Response Compensation and Liability Act
CIA – Controlled Industrial Area
CVAA – cold vapor atomic absorption
DL – detection limit
DOD – Department of Defense
ELISA – enzyme linked immune-sorbent assay
EPA – Environmental Protection Agency
ESTCP – Environmental Security Technology Certification Program
ETV – Environmental Technology Verification
GC/MS – gas chromatography/mass spectroscopy
ICP-MS – inductively coupled plasma – mass spectroscopy
lw – lipid weight
max - maximum
MCL – maximum cleanup level
MeHg – methylmercury
min - minimum
NAVFACNW – Naval Facilities Engineering Command Northwest
ND – not detected
OU B – Operable Unit B
PAC – powdered activated carbon
PAH – polycyclic aromatic hydrocarbons
PCB – polychlorinated biphenyl
PDMS – polydimethlysilozane
PRC – performance reference compound
PSNS & IMF – Puget Sound Naval Shipyard & Intermediate Maintenance Facility
ROD – Record of Decision
RSC – rapid screening characterization
SD – standard deviation
SDI – Swartz dominance index
SEA Ring – Sediment Ecotoxicity Assessment Ring
SPI – sediment profile imaging
SPME – solid phase micro extraction
SQC – sediment quality criteria
SSC Pacific – Space and Naval Warfare Systems Center Pacific
TOC – total organic carbon
ww – wet weight
XRF – xray fluorescence

INTRODUCTION

Successfully demonstrating the delivery, placement, and effectiveness of in-situ treatment materials in active harbors has the potential to reduce costs, shorten recovery times, and provide more effective alternatives to traditional methods of remediation for a wide range of sites with contaminated sediment. Although in-situ treatment is described in Environmental Protection Agency (EPA) guidance for remediating contaminated sediment sites (US EPA 2010), large scale demonstrations, implementation, and acceptance are generally lacking (Ghosh et al. 2011). Traditional sediment remedies usually involve removal by dredging or isolation by capping. However, removal actions may cause increased mobility and bioavailability of the contaminants, physical capping may not be practical in active harbors and navigable waterways, and dredging and capping may cause more harm than good (Ghosh et al. 2011). Significant challenges for amendment placement in active harbors include security access, scheduling, deep water placement, working near and under waterfront structures, complex bathymetry and dredge cuts in berthing areas, strong and variable tidal currents, and possible disturbance from ship movement and other harbor activities.

The overall objective of this project is to demonstrate and validate placement, stability, performance and persistence of reactive amendments for treatment of contaminated sediments in active Department of Defense (DoD) harbor settings. Specifically, this project has four technical objectives focused on 1) evaluating the ability to place the amendment in deeper water areas and near piers and structures that support vessel traffic, 2) evaluating the short-term (1 yr) effectiveness of amendment in controlling contaminant bioavailability, 3) evaluating the physical stability and longevity (persistence) of the amendment in the sediment (3 yrs), and 4) tracking the response of the benthic community to the amendment application. The technology that is being demonstrated incorporates a combination of an activated carbon (AC) reactive amendment, a conventional delivery system, and a suite of robust monitoring techniques for assessing delivery, stability, effectiveness in reducing bioavailability, and potential adverse effects.

The goal of this demonstration was to conduct a full scale (minimum 0.5 acre) amendment application at an active deep-water harbor location adjacent to Pier 7 at the Puget Sound Naval Shipyard & Intermediated Maintenance Facility (PSNS & IMF, **Figure 1**). Amending contaminated sediments with a chemical sorbent is designed to enhance ecosystem recovery by sequestering the contaminants thereby reducing uptake by sediment dwelling organisms and lessening the flux into the overlying water column. Recently, activated carbon has shown promising results at the pilot scale for reducing bioavailability of hydrophobic contaminants such as polychlorinated biphenyls (PCBs, Ghosh et al., 2011; McLeod et al., 2007; Millward et al.; 2005; Sun and Ghosh 2008), polycyclic aromatic hydrocarbons (PAHs, Cornelissen et al., 2006), mercury (Hg) and methylmercury (MeHg, Ghosh et al., 2011; Kim et al. 2011), dioxins (Fagervold et al., 2010), and other contaminants (Tomaszewski et al., 2007). However, most applications have used granulated AC which is not well suited to use in deep water due to its low density and difficulty in applying the amendment to the seafloor at depth. In this white paper, we summarize the results of Technical Objectives 1 and 2 above for the purpose of providing early communication of the short-term performance observed to date.

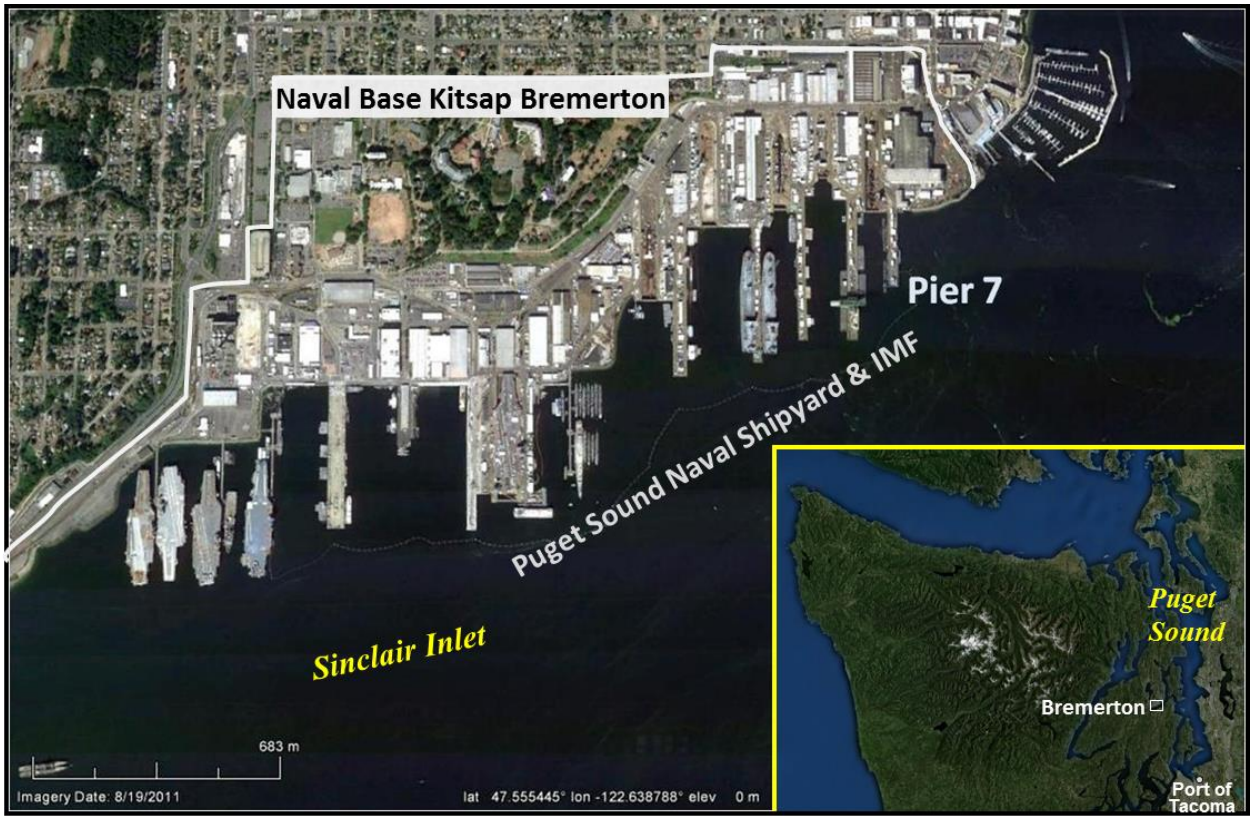


Figure 1. Location of Pier 7 at PSNS&IMF in Sinclair Inlet near Bremerton, WA.

Performance Objectives

Performance objectives for this project were specifically designed to assess physical endpoints (including placement, distribution, mixing and stability), chemical endpoints (including changes in PCB partitioning/sorption in the presence of the amendment), and biological endpoints (including tissue concentrations of contaminants and assessment of benthic community effects following placement). Data collected in support of these performance objectives will provide multiple lines of evidence for assessing the effectiveness of amendment placement as an in situ strategy for reducing chemical bioavailability at contaminated sediment sites. Performance is being analyzed using a combination of quantitative and qualitative tests (**Table 1**) to achieve the objectives of the project.

Site Background

Pier 7 is located inside the Controlled Industrial Area (CIA) of the shipyard and is part of the Bremerton Naval Complex (BNC) which includes Naval Base Kitsap Bremerton and PSNS & IMF. The shoreline is an industrial waterfront, armored with quay walls and riprap, with several large piers. Site characteristics for Pier 7 are shown in **Table 2**. Vessel traffic ranges from small recreational and commercial fishing vessels to occasional larger tug and Navy ship traffic, and regularly scheduled Washington State ferries arriving and leaving the Bremerton Ferry Terminal. Wind action in Sinclair Inlet generally creates a wave height range of 0.5 to 2.5 ft (0.15 – 0.76 m). Maximum wave heights are generated with winds from the SW (Wang and Richter 1999).

Table 1. Performance Objectives.

Performance Objective	Data Requirement	Success Criteria
Amendment reduction in contaminant bioavailability in the field.	Polycheate and bivalve bioaccumulation in situ with lab verification and passive sampler uptake to confirm reduction in porewater concentrations.	Reduction in biouptake of target CoC (PCBs) in treatment vs. controls. Target > 50% reduction.
Demonstrate reduction in contaminant bioavailability is sustained over time.	Polychaete and bivalve bioaccumulation *in situ*. Passive sampler uptake to confirm reduction in porewater concentrations.	Reduction in PCB bioaccumulation and uptake compared to baseline is sustained over 2 years. Hg and MeHg to be tracked.
Demonstrate uniform deep water placement to target footprint.	SPI images and cores within footprint and around perimeter of foot print.	Amendment is evenly distributed at the approximate target thickness (~2”). SPI camera able to distinguish amendment from native sediment and accuracy in identifying mixing depth within 50% of estimates indicated by carbon analysis of sediment cores.
Demonstrate amendment physical stability over time.	SPI images and cores within footprint and around perimeter of footprint.	Amendment remains evenly distributed laterally while mixing vertically over time.
Evaluate benthic community changes in response to amendment.	Benthic community census data.	< 20% adverse impact in benthic community ecological health metrics.

The sediments in the nearshore area of the shipyard have been designated as Operable Unit B Marine (OU B Marine) under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) response action for cleanup. A Record of Decision (ROD) for OU B Marine was signed in June 2000 (U.S. Navy, Ecology, and US EPA 2000). A component of the ROD required dredging contaminated marine sediments within OU B Marine and disposing them in a confined aquatic disposal (CAD) pit created within inner Sinclair Inlet. The remedy also included monitored natural attenuation, which relies on natural sediment recovery processes to gradually cover any residual contamination with cleaner sedimentary deposits. The objective of the remedy was to reduce sediment-bound PCB exposure to benthic infauna to protect tribal consumption of fish and shellfish. Cleanup goals for PCBs were defined for area-weighted average sediment concentrations and English sole fish tissue concentrations (U.S. Navy, Ecology, and USEPA 2000). Subsequent reviews identified that Hg was also a contaminant of concern for tribal consumption of fish and shellfish (NAVFACNW 2012). During a fender pile replacement project for Pier 7 in 2010, elevated PCBs, Hg, and other contaminants was found adjacent to Pier 7 (NAVFAC NW 2012). Based on these findings, the Space and Naval Warfare Systems Center Pacific (SSC Pacific) was tasked to perform a laboratory treatability study to test and evaluate an

alternative *in situ* sediment treatment method using a reactive amendment on sediments collected from Pier 7. Concurrently, the SSC Pacific submitted a proposal to the Environmental Security Technology Certification Program (ESTCP) to conduct a full-scale sediment amendment demonstration project at the site using activated carbon (Chadwick et. al 2011). The proposal was selected for funding in Fiscal Year 2011, and following a successful laboratory go/no-go evaluation (Kirtay et al. 2012), the field demonstration was initiated in August 2012 as a remedial action under the CERCLA ROD for OU B Marine.

Table 2. Pier 7 Site Characteristics for tide, depth, temperature, salinity, current speed and sediment texture expressed by percent gravel, sand, mud, clay, and total organic carbon (TOC).

Tide (NOAA 2012)	**Mean Range**	**Diurnal Range**
	2.44 m	3.58 m
Bottom Depth (NOAA 2011)	**Avg**	**Range**
	12.5 m	10.7 -15.5 m
Temperature (Alberston et al. 1993)	14.5 C	9.7 - 21.7 C
Salinity (Alberston et al. 1993)	29.3 PSU	28.3 - 30.3 PSU
Current Speed (Wang and Richter 1999)	**Avg**	**Upper Bound**
	2.5 cm/s	40 cm/s
Sediment Texture (McLaren 2011)		
Type	**Sandy Mud/Muddy Sand**	
	Phi	**mm**
Mean	4.35	0.05
	Avg	**Range**
Gravel %	0.0	0 - 0
Sand %	45.2	20.5 - 75.7
Mud %	54.8	24.3 - 79.5
TOC % (URS 2012)	**3.1**	**2.6 - 3.5**

Lab Treatability Study

Purpose

In 2011, SSC Pacific carried out laboratory treatability studies by mixing commercially available powdered activated carbon (PAC) reactive amendment AquaGate + PAC™ with PCB- and Hg-contaminated sediments obtained from the contaminated area adjacent to Pier 7 at PSNS & IMF. AquaGate + PAC™ is a patented, composite aggregate technology resembling small stones and typically comprised of a dense aggregate core, clay or clay-sized materials, and polymers. The formulation tested for this study incorporates approximately 2-5% PAC, 10% clay and the remaining fraction of aggregate where the primary reactive amendment is a powdered activated carbon (PAC) bound to the dense aggregate particle with appropriate clay minerals. Components of treatability study included pre-screening the site to delineate the nature and extent of contamination, conducting laboratory studies to verify the effectiveness of the amendment in terms of reducing contaminant bioavailability, and testing

the Sediment Profile Imaging (SPI) system (Germano & Associates, 2012) for its ability to distinguish the amendment from native site sediment to support monitoring the placement, stability and mixing of the amendment after installation.

Treatability Study Materials and Methods

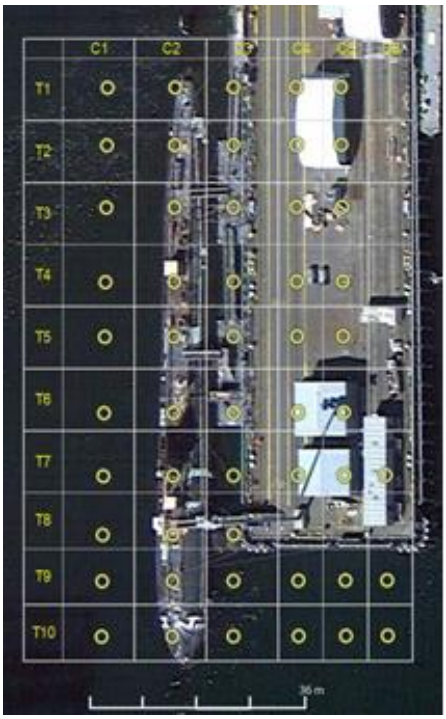
A diver assisted sediment survey was conducted around Pier 7 to more thoroughly delineate the nature and extent of contamination at the site and to identify sediments that could be used for the lab treatability study. Ten transects perpendicular to the pier were established with 4 in (10 cm) surface cores taken about every 50 ft (15 m) in the berthing area and every 30 ft (9 m) under the pier and avoiding the recently disturbed area 15 ft (4.5 m) on either side of the fender pilings (see **Figure 2**). Rapid Sediment Characterization (RSC, Kirtay et al. 2001) methods were used to rapidly screen the samples using a portable X-ray Fluorescence (XRF) detector for metals (Cu, Zn, and Pb) and Enzyme Linked Immuno-Sorbent Assays (ELISA) for PCBs (as Aroclor 1254, RaPID^{TM} Assay, Strategic Diagnostics Inc., Newark, DL) and polycyclic aromatic hydrocarbons (PAHs, as total PAHS, EPA Method 4035). A subset (20%) of the samples was used for confirmation analysis using more expensive analytical techniques including inductively coupled plasma mass spectroscopy (ICP-MS) for metals and gas chromatography/mass spectroscopy (GC/MS) for organics (Guerrero et al. 2011). A split of each sample was also submitted for laboratory analysis for total Hg using cold vapor atomic absorption (CVAA) and grain size distribution (McLaren 2011).

Bioassay experiments using site sediments amended with a standard formulation of the AquaGate + PAC™, as described above, were conducted to verify the effectiveness of the amendment material in terms of reduction in contaminant bioavailability to benthic organisms. The bioassay tests also evaluated different degrees of mixing including a No Mix, a Partial Mix (24 hour) and a Full Mix (1 month). Bioassay testing involved running standard 10-day amphipod and 28-day polychaete bioassays to assess any potential adverse toxic effects to growth and mortality endpoints from a) the native sediment, b) the uncoated aggregate that acts as the delivery mechanism for the AquaGate + PAC™, and c) the activated carbon-coated AquaGate + PAC™. Bioaccumulation testing involved running standard 28-day bioaccumulation studies on the reactive amendment/sediment mixtures. PCB sediment concentrations were also measured in each of the untreated and treated sediments used for the bioaccumulation studies. The aggregate was removed by the lab prior to homogenizing and analyzing the samples using standard methods (EPA method 8082).

Additional lab testing also involved evaluating the degree to which the Sediment Profile Imaging (SPI camera) system with digital image analysis was able to distinguish the amendment from native site sediment post-placement. The ability to monitor the placement and the physical stability of the reactive amendment in deeper water areas that support vessel traffic is a vital component in demonstrating the efficacy of this type of in situ treatment method. Testing was carried out pre- and post-application and mixing of the amendment, via mechanical means, to the native sediment.

Treatability Study Results and Discussion

Site Characterization: Within 36 hours of sampling, the screening data were used to identify the location of elevated PCB contamination. The results showed an isolated area of elevated contamination for PCBs and patchy locations of elevated total Hg (**Figure 2**). Bulk sediment samples were collected from cell (T6, C3) for the laboratory go/no-go evaluation study (Kirtay et al. 2012). Washington State Sediment Quality Criteria (SQC) and Maximum Cleanup Levels (MCL) (WAC 173-204) were exceeded by the maximum concentrations of PCBs, Hg, Cu, and Zn, while only Hg exceeded the sediment standards based on the ninetieth percentile (90%ofmean) of the geometric mean (geomean, **Table 3**). Sediment texture ranged from sandy mud to muddy sand with an average size of 4.35 ϕ (0.05 mm) and average TOC content of 3.1% (**Table 1**). Additional sediment was collected from the area where the highest levels of PCBs were measured (T6-C3) and shipped to SSC Pacific for the lab studies.



	C1	C2	C3	C4	C5	C6
T1	234	192	91	46	152	
T2	176	151	28	261	58	
T3	170	96	285	74	48	
T4	341	140	113	133	11	
T5	111	594	159	150	74	
T6	243	262	6650	305	439	
T7	224	261	129	655	193	105
T8	227	129	163			
T9	74	74	73	84	80	
T10	161	24	126	92	134	115

	C1	C2	C3	C4	C5	C6
T1		0.64	0.40	0.24	0.62	
T2	0.52	0.20		0.70	0.17	
T3	0.43	0.11	0.73	0.19		
T4	1.19	0.29	0.45	0.57	0.21	
T5	0.21	0.83	0.69	0.44	0.49	
T6	0.56	0.49	0.90	0.36	0.60	
T7	0.33	0.85	0.14	0.69	0.34	0.26
T8	0.12	0.17	0.28			
T9	0.32	0.35	0.17	0.06	0.18	0.20
T10	0.67	0.06	0.44	0.81	0.92	1.00

Figure 2. Sediment sampling transects at Pier 7 and the results for PCBs (ELISA; ng/g) and Hg (CVAA; μg/g).

Lab Bioaccumulation, Toxicity and Sediment Chemistry: The results from the treatability study demonstrated that amending the contaminated sediment collected from the Pier 7 site with activated carbon (in the form of AquaGate + PAC™ used in this study) effectively reduced the bioavailability of PCBs to the marine polychaete, *Neanthes arenaceodentata*. Increasing AquaGate contact time with the Pier 7 sediment resulted in progressively lower bio-uptake with up to 94% reduction of total PCBs for the 1 month mixed treatment (**Figure 3**).

Table 3. Site Characterization Results for PCBs, PAHs, Cu, Zn, Pb and Hg showing the detection limit (DL), minimum (min), maximum (max), average, standard deviation (SD), geometric mean (geomean), ninetieth percentile of the geomean (90%ofmean) and corresponding Washington State Sediment Quality Criteria (WA-SQC) and Maximum Cleanup Level (WA-MCL) standards.

	PCBs ng/g	PAHs µg/g	Cu µg/g	Zn µg/g	Pb µg/g	Hg µg/g
DL	44.7	0.18	70.1	60.7	39.7	0.005
min	4.6	0.18	70.1	60.7	39.7	0.058
max	5549.1	4.58	3659.1	1182.7	431.6	1.189
averae	241.1	1.32	176.2	273.2	83.5	0.445
SD	773.1	0.75	507.6	188.1	61.7	0.278
geomean	101.5	1.12	100.9	230.7	73.1	0.356
90%ofmean	288.8	2.09	199.6	396.5	114.8	0.738
median	112.1	1.38	70.1	228.0	61.0	0.402
WA SQC	372.0*	41.23*	390.0	410.0	450.0	0.410
WA MCL	2015.0*	188.48*	390.0	960.0	530.0	0.590

* Assuming a TOC content of 3.1%.

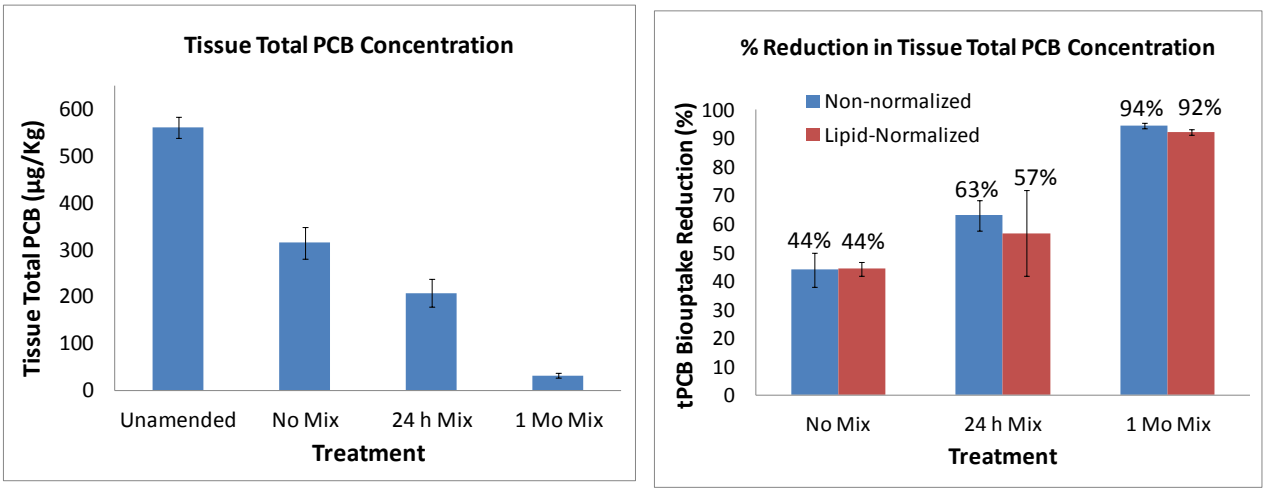


Figure 3. Tissue (wet weight) total PCB concentration (left) and percent total PCB tissue concentration reduction (right) in *Neanthes arenaceodentata* following 28-day laboratory exposures following different mixing duration of Pier 7 sediment amended with AquaGate + PAC™. N= 3 replicates per treatment.

Laboratory toxicity testing was also carried out to assess any potential adverse toxic effects from the unamended as well as amended sediment. Polychaete survival was very high with ≥ 96% survival in all treatments. Growth was not adversely affected when compared to the control sediment for any treatment, nor when the unamended sediment was compared to the 1 Month Mixed treatment ($p>0.05$). However, the No Mix and 24-Hr Mix treatments did result in statistically lower final weights relative to the unamended Pier 7 sediment (**Table 4**). These results suggest that there is an increased likelihood for reduction in growth immediately following amendment addition, possibly due to a more concentrated, less homogeneous exposure of the PAC to the polychaetes initially. However, over time growth does not appear to be adversely affected.

PCB sediment concentrations were also measured in the unamended and amended sediment samples at the end of the experiment. The results showed a fairly dramatic decrease in total PCB concentrations between the unamended and the amended sediments (**Figure 4**) which could be attributed to a change in the extractability of the PCBs from the unamended sediments and the sediments amended with the activated carbon.

Table 4. Treatability Study Results from 28-day Exposures with the Marine Polychaete *Neanthes arenaceodentata*. N=9 for Survival and Wet Weight; N=3 for Lipid Data. Statistical Differences among Treatments are Indicated by Different Letters (p<0.05).

Sample ID	Survival (%)			Individual Wet Wt. (mg)			Lipid (%, wet wt.)		
	Mean	s.d.	Sig.	Mean	s.d.	Sig.	Mean	s.d.	Sig.
Control	96	2.7	A	19.6	2.0	A	1.7	0.51	A
Unamended	97	5.1	A	23.5	3.0	B	2.0	0.15	A
No Mix	97	3.9	A	18.4	1.4	A	2.0	0.13	A
24 Hr Mix	96	5.0	A	18.9	1.1	A	1.9	0.69	A
1 Mo Mix	97	3.2	A	22.5	2.7	B	1.4	0.06	A

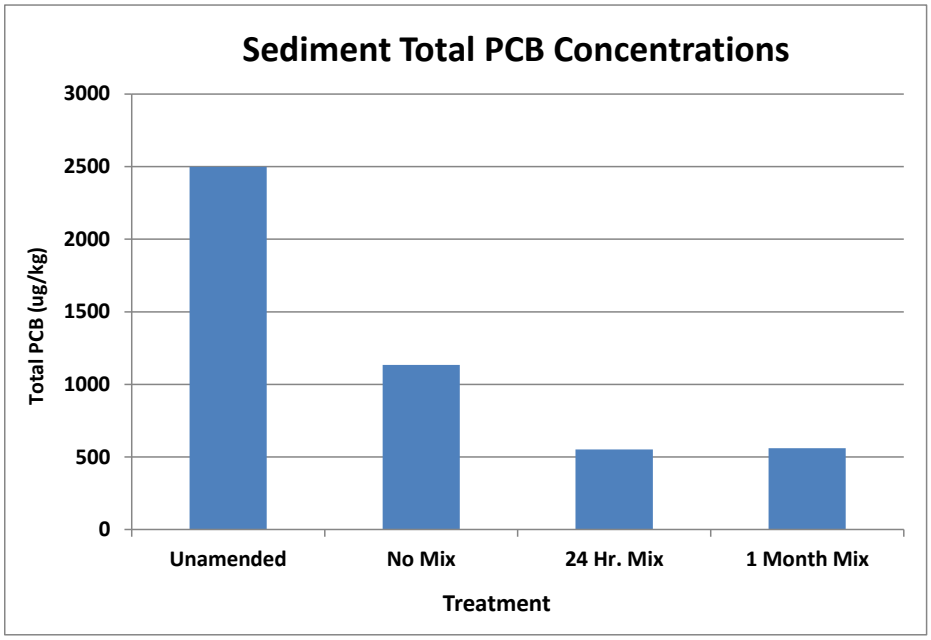


Figure 4. Total PCB sediment concentrations (µg/kg) in the unamended and amended (no mix, 24-hr mix and 1-mo mix) sediment samples used for the lab bioaccumulation studies.

Verification of Amendment Placement and Mixing: A hand-held Sediment Profile Imaging system (SPI camera) was tested in the laboratory on four different sediment treatments: 1) unamended sediment, 2) sediment with 1" layer of AquaGate + PAC™ placed on top, 3) amended sediment with a 1: 1 mixture of sediment and AquaGate + PAC™ placed on sediment surface and 4) Amended sediment with a 3: 1 mixture of sediment and AquaGate + PAC™ placed on sediment surface. In each of the amended treatments, two distinct layers could be observed in the SPI images. While the distinction between the amended layer and the underlying sediment was less obvious in the 1:1 and 3:1 mixtures as compared

to the initial treatment (AquaGate + PAC™ placed on sediment surface), the amended layer could be distinguished as a thicker, darker, more consolidated layer on top of the coarse-grained sediment below (**Figure 5**).

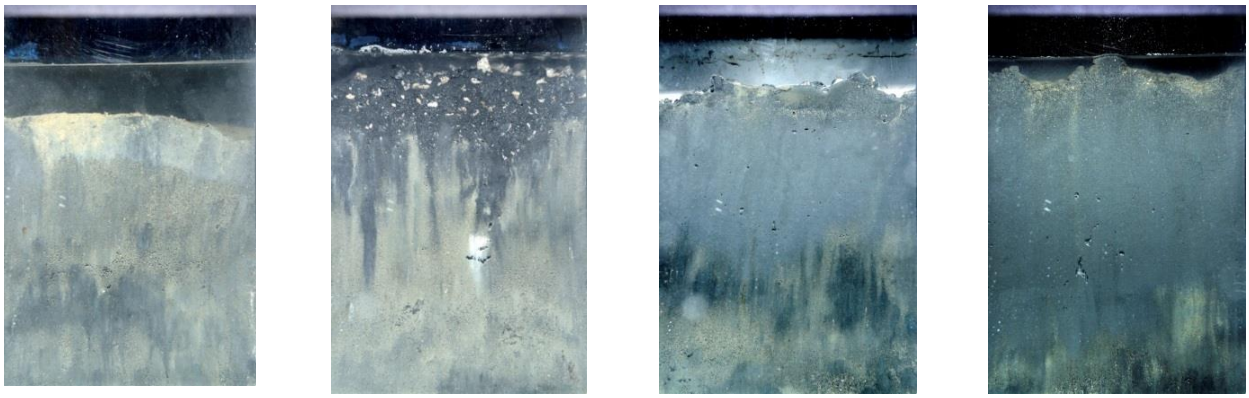


Figure 5. SPI camera images from 1) unamended sediment, 2) AquaGate+ PAC™ on top, 3) 1:1 mixture and 4) 3:1 mixture, left to right.

The results from this study suggested that the formulation of AquaGate tested in the lab would be effective at reducing the PCB bioavailability. Additionally, the results from testing the SPI camera for as a placement/stability verification monitoring tool also yielded promising results as each tool was able to distinguish the amendment from the native sediment. Results from the lab treatability study were used to support the design of the full-scale demonstration at Puget Sound Naval Shipyard & Intermediated Maintenance Facility (Kirtay et al. 2013).

REACTIVE AMENDMENT INSTALLATION

Reactive Amendment

AquaGate + PAC™ (AquaBlok Ltd, Toledo, OH) is made of composite particles manufactured with a crushed stone core coated with a combination of bentonite-based clay and PAC. The formulation used for this project was 2-5% (by weight) PAC, 5-10% clay (calcium bentonite) and 85% aggregate (60 – 96 mm), with a dry bulk density of 85 - 90 lbs/ft^3 (1.36 -1.44 g/cm^3). After placement on the sea floor, the PAC is designed to separate from the aggregate core and become incorporated into bottom sediment. The product was manufactured in Toledo, OH and transported to the Port of Tacoma, WA in seven truck-hauled containers containing the product packaged in water resistant, 2,400 lb (1095 kg) weight, "Super Sacks" (bulk bags), where the material was staged on a barge for transport to the shipyard. Because, the product is sensitive to water, care was taken to protect the material from water damage at all times.

Product Placement

On October 15, 2012, both the tug MARGARET MARY and barge ABERDEEN arrived on site at Pier 7. Amendment placement commenced at 2000 on October 16 and the contractor worked 10-12 hour shifts through the nights of October 16-17 to take advantage of the favorable tides and weather. The product was placed in both open berthing and under pier areas from the tug-operated, moored barge that contained the staged product packaged in the "Super Sacks," a backhoe loader that moved each bag to a hopper feeder, and a truck-mounted conveyor belt-type (telebelt) broadcast conveyor system (**Figure 6**).

The broadcast application obtained a rapid, relatively uniform placement of about 143 tons (130 metric tons) of product over the target area. The equipment was able to place the product both in the open access berthing area and under the pier by accessing the under pier areas between existing pilings during low tide. Most of the placement occurred at night to accommodate the tidal conditions. Measurements of the amendment thickness were made by placing a 5 gallon bucket on the seafloor next to the pier and capturing the product as the conveyor distributed the product along the pier. About 2 – 4 in (5 – 10 cm) of the product was captured in the bucket from the single pass used to distribute the product. Once the barge was moored in the desired position, distribution of the product occurred relatively quickly, resulting in cycle-time of about 3 min/sack to distribute the product.

On the morning of October 19, the PSNS&IMF Divers were on site to observe placement of the final two sacks during daylight hours. Diver observations of product delivery showed that the small pebbles were resistant to the current velocities and sank slowly to the bottom settling on the existing bottom substrate, without any untoward impact to sea life on the bottom, such as sea anemones, sea stars, crabs, and flat fish. No turbidity plumes associated with the placement were observed. Diver surveys, sediment cores, and SPI camera monitoring of the placement area conducted on Oct 30-31, 2012 showed that the PAC covering had released from the aggregate, as the light-colored aggregate was plainly visible on the seafloor (**Figure 7**). Initial observations indicated that the amendment was placed effectively over the target area; however some variation in thickness was observed and there was some overspray or drift of the amendment slightly beyond the edge of the target area.

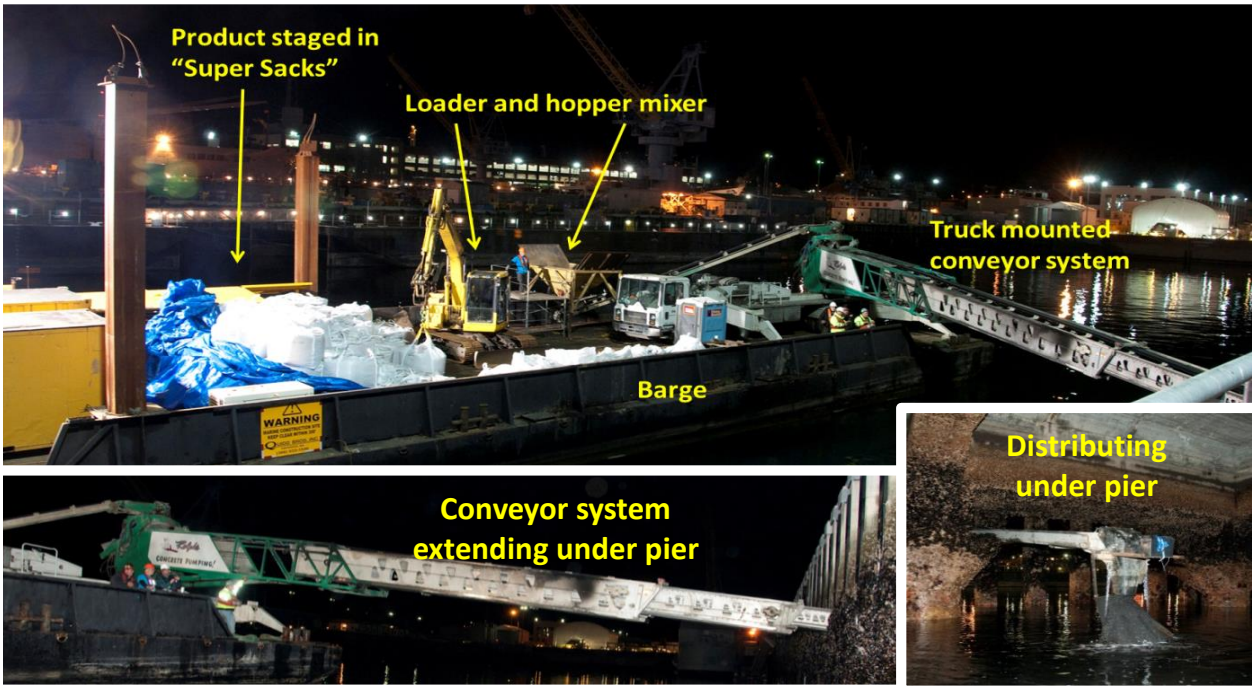


Figure 6. Product placement (images cleared for public release).

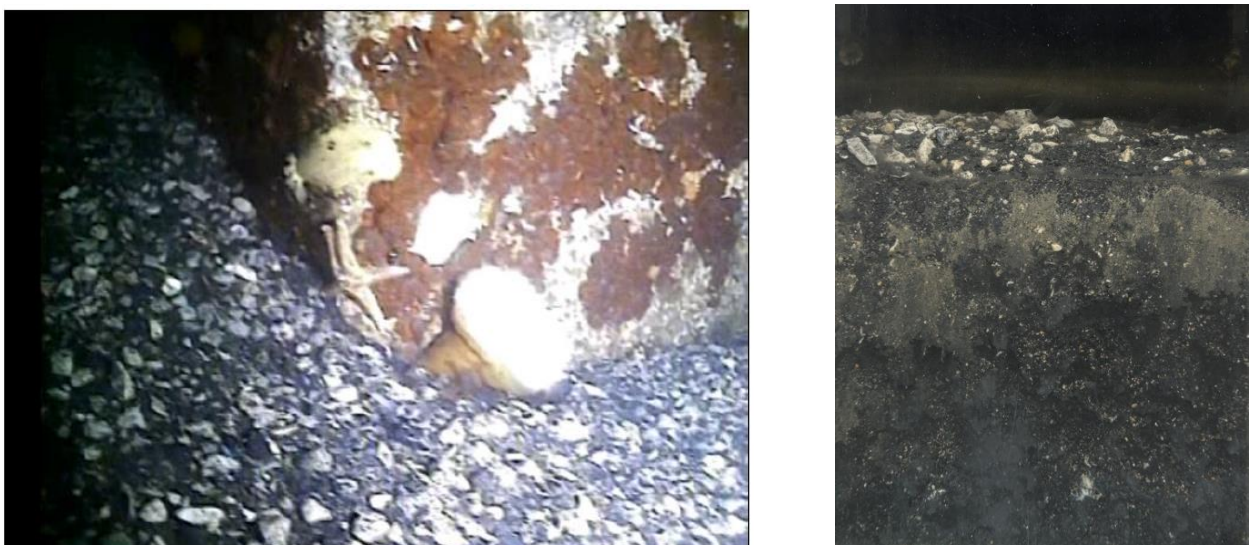


Figure 7. Diver survey and SPI camera images 2-weeks post product placement. Aggregate plainly visible on seafloor (images cleared for public release).

MONITORING METHODS

Experimental Design

The demonstration was designed to provide baseline (pre-amendment placement) monitoring and post-amendment placement monitoring at several intervals over a three year period. Several physical, chemical, and biological parameters are being used to evaluate the success of placement and stability of amendment and assess the effectiveness of the reactive amendment (AquaGate + PAC™) to reduce bioavailability following placement at 0.5, 3, 10, 22 (underway) and 34 (planned) months.

The physical parameters assessed in this project included evaluating the distribution, coverage, uniformity, and thicknesses of the amendment immediately after placement; the stability of the amendment and changes in amendment stability over time resulting from natural sedimentation, benthic mixing, ship and tug activity; increased sediment cohesiveness over time; and visual monitoring with the SPI system to evaluate amendment presence, thickness, and mixing depths. Chemical parameters included monitoring the surface sediment chemical concentrations prior to and following amendment delivery to evaluate changes in bulk concentrations and monitoring of the reduction in contaminant bioavailability (magnitude and sustainability of the bioavailability reduction). Biological parameters included assessing the reduction of PCBs in the tissues of clams and worms and assessing changes in benthic community richness, abundance, distribution, evenness, and diversity before and after placement.

Dive Support

Dive support for the project was provided by the PSNS & IMF Dive Locker. The divers were equipped with SuperLite® 17 helmets (Kirby Morgan Dive Systems, Inc., Santa Maria, CA) and neoprene ½ in wet suits with surface supplied air and warm water through an umbilical tether system from the dive boat. The dive team consisted of two divers, two tether handlers, a dive supervisor and backup, standby divers. The divers were in constant communication with the dive supervisor and scientific team with an audio communications system and an underwater video camera (UWS-3200, Outland Technology, Slidell, LA) with a light emitting diode (LED) that was either attached to the diver's helmet or hand held. The video was displayed on a monitor onboard the dive boat and the video and audio from the divers were recorded with a digital video recording (DVR) device. The direct communication with the divers was very valuable to the scientific team, as the divers were able to communicate information about sea floor conditions and provide feedback on amendment placement as well as equipment performance and sampling conditions during each monitoring event.

Methods and Tools

To date, field monitoring events have been conducted 2 months before (baseline) and 0.5, 3, and 10 months after the activated carbon amendment was applied in October 2012 (**Figure 8**). Several different technology components and methods were used including the sediment ecotoxicity assessment benthic chamber ring (SEA Ring) device for in situ bioaccumulation measurements, petite Ponar grab sampler to collect benthic invertebrate census samples, sediment coring for measurements of sediment chemistry, black carbon and total organic carbon content, in situ SPME passive sampler to

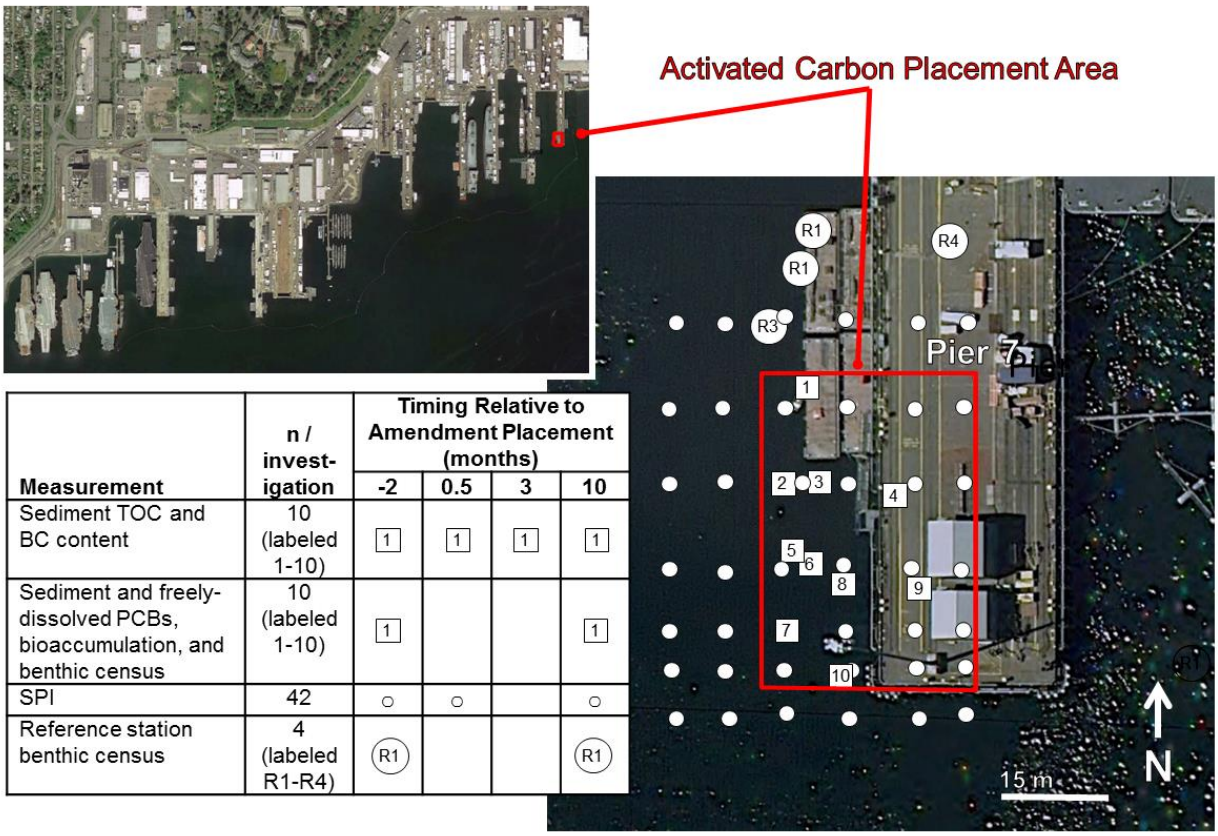
measure concentrations of PCBs in sediment porewater and SPI camera to provide a qualitative examination of the benthic community and to assess the depth of sediment mixing via bioturbation. Detailed information regarding each of the individual measurements is provided below.

Surface Sediment Cores

In all four investigations, several 5-cm diameter plastic core tubes were inserted by divers into sediment at each of the ten numbered stations (**Figure 8**), capped manually, and returned to the surface for processing. Cores for chemical analysis were split lengthwise in order to obtain samples of the discreet sediment layers at 0-5 cm, 5-10 cm, and 10-15 cm below the sediment-water interface following removal of large (> 0.5 cm) debris and aggregate. Samples of the discrete intervals were analyzed for TOC, black carbon (BC), mercury, methylmercury, and PCBs. Samples were stored at 4°C until analysis. At the 10-month investigation, cores collected for organic carbon and chemical analysis were also inspected visually during sample collection, and aggregate presence or absence was noted for the 0-5, 5-10, and 5-15 cm sediment layers. During all four investigations, samples were analyzed for TOC and BC via the Lloyd Khan method. At the baseline and 10-month investigations, concentrations of PCB congeners were quantified using EPA Method 8082 and mercury and methylmercury by QS-LC-CVAF-003 (QuickSilver Scientific) method [Note: at this time results for mercury and methylmercury are not available therefore they are not included in the paper]. During the baseline and 10-month investigations, six core samples per station were also collected for benthic macroinvertebrate census. The area sampled by the six cores corresponded to a sampled area of 0.01 m^2. At each station, the six cores were combined, stored at 4°C briefly (24-48 h), and sieved (500-µm) to obtain macroinvertebrates, which were preserved, identified to the most specific taxon, and enumerated.

Measurements of PCB Bioaccumulation

During the baseline and 10-month investigations, measurements of bioaccumulation by worms and clams were made using the SEA Ring. Consisting of an autonomous multi-chamber benthic sampler, the SEA Ring is a patented (U.S. Patent No. 8,011,239), integrated, versatile, field tested, toxicity and bioavailability assessment device (Burton et al. 2013, Rosen et al. 2012) that has successfully completed EPA's Environmental Technology Verification (ETV) Program (Darlington et al. 2013). On deployment day, five *M. nasuta* clams (1 in in size) collected from a suitable reference site were loaded into 4 replicate exposure chambers on the SEA Ring that were enclosed with coarse (1/2" stainless steel) mesh. Four other replicate chamber were used to deploy a total of 20 five-week old *N. caecoides* polychaete worms collected from the reference site by loading 5 worms into the 30 mL syringes embedded in each of the SEA Ring chamber caps for later release into the open bottomed sediment chambers following placement at the site. Each SEA Ring was prepared for deployment while held in a 17 gallon Chemtainer (Chem-Tainer Industries, chemtainer.com) filled with site water that was lowered into the water where divers removed the SEA Ring and deployed the unit on the sea floor at the desired sample location by gently inserting all the chambers into the bottom until the base of the unit was flush with the sediment surface. After securing the SEA Ring to the bottom with stakes and marking the site with a submerged, clearly-labeled buoy, the divers released the worms by depressing the syringe plungers.



Measurement	n / invest-igation	Timing Relative to Amendment Placement (months)			
		-2	0.5	3	10
Sediment TOC and BC content	10 (labeled 1-10)	1	1	1	1
Sediment and freely-dissolved PCBs, bioaccumulation, and benthic census	10 (labeled 1-10)	1			1
SPI	42	○	○		○
Reference station benthic census	4 (labeled R1-R4)	R1			R1

Figure 8. Location of measurements made 2 months prior (baseline) and 0.5, 3, 10 months after the activated carbon amendment was applied at Pier 7, PSNS & IMF, Bremerton, Washington, USA.

Following the 14 day exposure period, the SEA Rings were recovered by divers. Following an initial visual assessment of each SEA Ring, stakes were removed, polyethylene end caps were affixed to the bottom of each exposure chamber prior to removal from the sediment, and the device was gently lifted out of the sediment after activating the SEA Ring vacuum recovery system to prevent loss of sediment from cores. The SEA Ring was then placed into the Chemtainer and transferred to the boat crew. Polychaetes were recovered from replicate chamber by extruding the contents onto a 500 µm stainless steel sieve and washing with seawater pumped from the site to retain the organisms. Clams were recovered from the sediment by hand. Organisms were then transported to a laboratory where they were placed in clean seawater, depurated for 24 hr, and then prepared for shipping on ice to the analytical laboratory for analysis. Upon receipt, the analytical laboratory kept all specimen samples frozen until they were homogenized as composites of individuals from each chamber for analysis of PCB congeners, total mercury, methylmercury, and lipid content.

Successful retrieval of *M. nasuta* was not obtained in the baseline investigation for station 6. As part of the effort to validate the performance of the SEA Ring (Rosen et al. in prep), *M. nasuta* were exposed to intact sediment cores from each station for two weeks using the same SEA Ring chambers in a laboratory flow-through system during the same time periods that the SEA Rings were deployed in situ. Because SEA Ring bioaccumulation results from the laboratory and field SEA Ring tests were similar for other stations and sediments (Rosen et al. in prep), the laboratory results from station 6 were used as a

substitute and combined with other field data from the baseline investigation. At stations 1, 2, 7, and 10 in the baseline study, and stations 2, 3, 5, 6, and 8 in the 10-month study, *N. caecoides* were not recovered. Laboratory results for SEA Ring tests with *N. caecoides* were available for stations 5 and 6 for the 10-month investigation, and this data was used a substitute and combined with other field data from the 10-month investigation.

Measurements of PCBs in Sediment Porewater

Measurements of tri-, tetra-, penta-, and hexachlorinated biphenyl congeners in sediment porewater were conducted by inserting samplers containing SPME fibers into surface sediment at the ten numbered locations shown in Figure 8. The SPME method was adapted from procedure of You et al. (2007), Yang et al. (2008), Lu et al. (2011), Oen et al. (2011), and Harwood et al. (2012). Each SPME sampler consisted of twelve 12.5-cm pieces (150 cm total) of SPME fiber (10-µm thickness polydimethylsiloxane (PDMS) coating, 210-µm silica core diameter, (Fiber-guide Industries, Stirling, NJ). The fibers were contained in within a 110-µm stainless steel mesh envelope, cleaned with a 50:50 solution of acetonitrile:water and water rinse, and pre-loaded with performance reference compounds (PRCs) by exposing SPMEs for 1 d in an end-over-end-mixed, 80:20 methanol:water solution containing PCB-29 (Trichlorinated PRC), PCB-69 (Tetrachlorinated PRC), PCB-104 (Pentachlorinated PRC), and PCB-154 (Hexachlorinated PRC) at concentrations of 0.2 µg/mL. At each of the ten stations, one envelope as was attached to one of the SEA Ring cylinders and one was attached to a disposable plastic core tube such that when the SEA Ring and core tube were inserted into the sediment by divers, the SPME fiber was exposure to the top 10 to 15 cm of surface sediment.

Although the divers attempted to insert the entire length of the SPME sampler into the sediment, this was difficult due to the sediment strata, shell hash, lack of visibility, etc. At many of the stations, only a portion of the SPME was exposed to the sediment, with the remainder exposed to the sediment-water interface and overlying water. Upon retrieval of the SPMEs core tubes and SEA Rings, the proportion of the SPME envelope buried in the sediment was recorded. On average, 62% (SD $\pm$25%) of the envelopes were buried beneath the sediment surface, indicating that results best reflect freely-dissolved PCBs in the top 7-8 cm of sediment and lower 4-5 cm of surface water (surface-water interface).

Upon recovery, envelopes containing SPMEs were individually wrapped in aluminum foil, placed in plastic bags, and stored at 4°C until they were shipped to the analytical laboratory where they were removed from the envelope, wiped with a moist tissue, cut into small pieces, placed in a 2-mL vial, and submerged in 1.8 mL hexane. The vials were then stored at 4°C for several days, spiked with an internal recovery standards (PCB-34, PCB-165, and PCB-209), evaporated to a volume of approximately 100 or 200 µL with pure nitrogen, and analyzed for PCB congeners consistent with EPA Method 1668 procedures.

In addition to SPMEs exposed to sediment, several trip blanks were also shipped to the site and extracted to provide concentrations of PRCs in the fiber that was not exposed to sediment. This data was treated as the initial concentration of PRCs in the PDMS and was used to adjust the PCBs measured in sediment-exposed SPME to steady state concentrations. This was accomplished by first calculating correction factors for each of the PRCs in each fiber (Oen et al., 2011) as inferred by the percentage of

steady reached, which was 68% (22%), 51% (20%), 40% (15%), and 21% (14%), on average (SD), for the trichlorinated, tetrachlorinated, pentachlorinated, and hexachlorinated PRC, respectively. Log10-transformed correction factors were regressed on their respective PDMS-water partition coefficients (Smedes et al., 2009) for the four PRCs, and the resulting model was used to calculate correction factors for each of the PCB congeners absorbed by the fibers exposed to sediment using PDMS-water partition coefficients. These correction factors were multiplied by the measured concentration of PCBs in the PDMS of fibers exposed to the sediment and divided by the respective PDMS-water partition coefficient to calculate the concentration of freely-dissolved PCBs in sediment pore water.

Sediment Profile Imagery

The SPI camera system was used to evaluate the placement and thickness of the amendment over the target area as well as to visualize physical processes at the amendment-water and sediment-amendment interface during the baseline, 0.5- and 10-month post-placement investigations. The SPI surveys also provided additional information on sediment characteristics including buried organic-rich horizons, depth and extent of biological mixing, and large-scale variations in sediment grain size that may indicate significant variations in energy regime. During the baseline, 0.5-, and 10-month post-placement investigations, SPI surveys were conducted using both a diver-deployed (handheld) and crane-deployed SPI cameras. The handheld unit was deployed by divers at 18 locations located either under the pier or near the pier pilings and the crane-deployed camera was used at 23 locations within the berthing area (**Figure 8**). During the 10-month SPI survey, eight additional stations were added to better delineate the edge of the reactive amendment cap.

While replicate images were taken at each station, the amount of disturbance caused by the diver-deployed system did not allow for reliable measurements of precision between replicate images, so only one replicate (the least disturbed) from each station sampled by divers was analyzed. The amount of debris, shell hash, and gravel placed during the piling removal project that was present in and around the piers created high variation in the penetration depth at the crane deployed stations, with cross-sectional sedimentary structures masked or destroyed by debris (natural or anthropogenic) being dragged down by the prism cutting blade. Given the variation in image feature preservation (regardless of whether they were taken with the crane- or diver-deployed system), and because this variation in cross-sectional structural appearance was not really indicative of natural variance in the measured parameters, the best image (least disturbed) from each station was selected for the analysis.

Benthic Community Health

To evaluate benthic community changes in response to placement of the amendment a benthic community census was conducted during the baseline and 10-month surveys. The baseline benthic community was sampled on August 14-15, 2012, and the 10-month post remedy sampling event was conducted on August 7-8, 2013. In both sampling events, ten surface sediment samples, co-located with the SEA Ring stations within the target amendment area and four reference stations outside the target amendment area, were collected and analyzed to identify the lowest possible taxonomic level of benthic invertebrates (**Figure 8**). Six biological indices commonly used to assess benthic community health were used to evaluate the data, including total abundance; species richness; species diversity, as measured by

Shannon-Wiener Diversity Index (H'); species evenness, as measured by the Pielou's Evenness Index (J', Pielou 1966); species dominance, as measured by Swartz's Dominance Index (SDI, Swartz et al. 1985); and dominance of the five most abundant taxa, as measured by the percentage of total abundance comprised of the five most abundant taxa. To evaluate potential impacts the remedy may have on the benthic community, the 10-month sampling event results were compared with the baseline. Statistical test procedures included t-tests, and nonparametric (Wilcoxon) tests on untransformed and log-transformed data, with a significance level of 0.05.

SPI camera images were also used as additional measures of benthic health before (baseline) and after amendment placement (T=0.5 months and T=10 months). The SPI camera was used to evaluate infaunal succession. These stages are recognized in SPI images by the presence of dense assemblages of near-surface polychaetes and/or the presence of subsurface feeding voids; both may be present in the same image.

Data Treatment and Statistical Analyses

To evaluate differences between baseline and 10-month concentrations of PCBs in *M. nasuta* and *N. caecoides* tissues, concentrations of PCBs on a lipid weight (lw) basis were used. Lipid content of organisms strongly influence bioaccumulation potential (Burkhard 2009), and enable a comparison of bioavailability among organisms with differing lipid contents. The latter issue was relevant in our study, because mean lipid content of *M. nasuta* used in the baseline investigation (0.0072 g lw/g ww) was a factor of 1.7 times lower (P = 0.004) than the lipid content of the clams used in the 10-month investigation (0.012 g lw/g ww). Lipid content of *N. caecoides* did not differ (P = 0.14) between the baseline (mean of 0.009 lw/g ww) and 10-month (mean of 0.011 lw/g ww).

Bioaccumulation test results between the baseline and 10-month investigations were compared on the basis of the sum of all measured PCB congeners (total PCBs) or the sum of congeners measured in each homologue group for tri-, tetra-, penta-, and hexachlorobiphenyl. In addition, an evaluation of commonly-detected congeners in tissue (e.g., PCB070, PCB091, PCB118) confirmed the overall conclusions reached by comparing the sums. Sums of homologues or congeners were based on detected PCBs; the concentration of congeners with a non-detect result were assumed to have a value of zero. In the single case in which all congeners were below the detection limit (or all congeners in a homologue group were below the detection limit), the highest detection limit of the congeners for the particular sample was used to impute a substitute value that was generated via Regression Order Statistics (ROS). ROS-generated substitute values were calculated by ProUCL (US EPA, http://www.epa.gov/osp/hstl/tsc/software.htm) assuming a lognormal distribution of values, and that non-detected values were greater than zero and less than the detection limit. The ROS method was selected because there were many non-detected valuess among some of the homologue groups (i.e., frequency of detection was ranged from 14 to 30%).

The concentrations of freely dissolved total PCBs in sediment pore water were expressed by summing detected tetra-, penta-, and hexachlorobiphenyl congeners. If no congeners were detected, the maximum detection limit of the tetrachlorinated congeners (0.02 – 0.4 ng/L) for the sample was used to

generate an estimate using the ROS procedure as described above. The frequency of non-detected values for the SPME samples was 20% and 75% for the baseline and 10-month surveys, respectively. Other non-detect data treatment methods (assuming values of 0, one-half the detection limit, and detection limit for non-detect results and evaluating only samples with detections) were used in sensitivity analyses, but overall conclusions did not differ from those reached using the data approach described above.

At several stations, only a portion of the SPME was buried beneath the sediment, so we assumed that PCBs were absorbed into the fiber from direct contact with the sediment. The concentration of PCBs in pore water was estimated by dividing the PCB concentration by the proportion of the fiber that was below the sediment surface. This conservatively assumes that the concentration of freely-dissolved PCBs in the overlying water (approximately 6 cm) above the sediment surface was zero. Although at least one experiment with passive samplers has confirmed this assumption (Fernandez et al., 2014), other experiments have suggested that concentrations of PCBs in near-sediment overlying water are similar to that of sediment porewater (Booij et al., 2013). The effect of our data treatment in this way was considered a bounding exercise, results of the freely-dissolved concentrations corrected for the proportion of the SPME below the sediment surface were roughly twice the uncorrected values. However, the sensitivity analysis with the corrected values did not result in different conclusions. For example, concentrations of freely-dissolved PCBs in the baseline were significantly different from those measured in the 10-month investigation using either uncorrected or corrected values, or the magnitude of the difference between baseline and 10-month data was nearly identical (5% difference). Therefore, results and conclusions are based on the uncorrected values.

Statistical comparisons of data collected during the baseline and the 10-month investigation were made using pooled t-tests or nonparametric tests (Wilcoxon rank sums) depending on the normality of the data and heterogeneity of variance, as tested by Shapiro-Wilks and Levene's tests, respectively. Because data often varied by several orders of magnitude and were log-normally distributed, data were Log10-transformed to improve statistical power. Statistical differences detected at the 0.05 level ($p \leq 0.05$) were considered significant.

RESULTS AND DISCUSSION

Verification of Amendment Placement and Short-term Stability

One of the primary performance objectives of this project was to demonstrate the ability to place the reactive amendment in a deep-water, active harbor environment as well monitor it's stability over time. Sediment profile imagery, visual inspection, and chemical/physical measurements (e.g., TOC and black carbon) of sediment core samples were used to evaluate this objective.

SPI: The SPI camera images taken during the baseline, 0.5-, and 10-month surveys clearly showed the presence of the AquaGate + PAC™ particles on the sea bottom (**Figure 9**). Even though the SPI survey was conducted only two weeks after the material had been placed, the covering of activated carbon particles had already dissolved off the underlying carrier granules (leaving what looked like white gravel on the sediment surface; see **Figure 10**). By 10 months the activated carbon was being worked into the underlying sediment by the burrowing activities of resident infauna and other mixing processes (Germano & Associates, 2013).

The SPI surveys showed that the placement of the activated cap amendment did not cover all of the originally targeted placement area with the AquaGate+PAC™ material. Measureable deposits of AquaGate+PAC™ could be seen at 12 stations, while 7 stations showed only traces of the AquaGate+PAC™ particles in the upper oxidized layer of sediment Approximately 75% of target area received at least a trace of amendment and approximately 70% of the target area received target thickness (5 cm) or more (**Figure 11**).

Some notable changes in both seafloor conditions and amendment placement and thickness were observed in the 10 month SPI survey. As noted from the October 2012 survey, right after the capping operation, the addition of the AquaGate+PAC™ amendment provided some surface armoring at select stations which impeded camera prism penetration. During the 10 month survey there was a noticeable decrease in prism penetration depth at the stations along the western edge of the pier compared with the results from October 2012. This likely caused by the cobble armoring (**Figure 12**) placed at the site during the fender pile replacement in 2010, when a "sand blanket" was placed along the fender pilings extending 15 ft on each side of the pilings. The differences in the SPI images from SPI station 1-3 south to SPI 4-3 (Figure 8) can be explained by slight variation in the location of the sample, the images obtained during October 2012 were probably a few feet farther west off of the "sand blanket". Measureable deposits of AquaGate+PAC™ could be seen at 16 stations, while 5 stations showed only traces of the AquaGate+PAC™ particles in the upper oxidized layer of sediment (**Figure 13**). While the overall percent coverage was similar to the 2012 survey (5 cm or greater over ~75% of the target area), the area in which the amendment was detected appears to have shifted south, with amendment detected at locations where it was not detected in the 2012 survey (**Figure 13**). While there were some substantial changes to seafloor conditions observed in August 2013 compared with those from October 2012 (Germano & Associates 2013), visual evidence of the reactive amendment being re-worked into the bottom sediments by bioturbation was clearly evident (**Figure 14**).

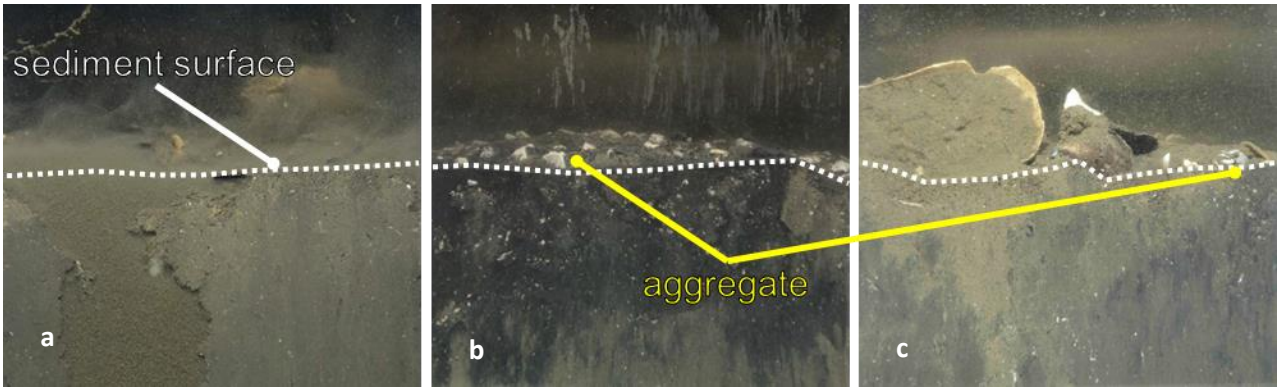


Figure 9. Representative Sediment Profile Images of the upper 8 cm of sediment at a SPI station located between SEA Ring stations 2 and 3 obtained during the baseline (a), 0.5-month (b), and 10-month (c) surveys. The width of each image is 14.5 cm (images cleared for public release).

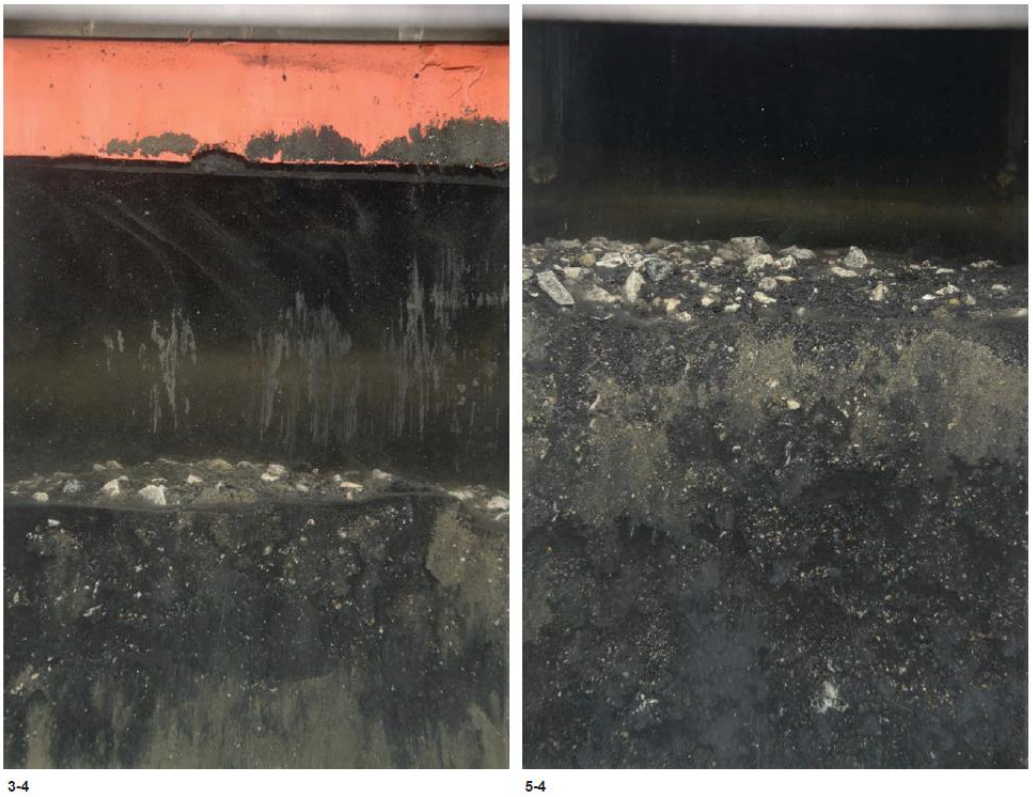


Figure 10. Sediment profile images from SPI station 3-4 (left) and 5-4 (right) show how the activated carbon covering on the particles for the AquaGate + PAC™ has dissolved off the carrier granules, leaving a surface armoring of white pebbles while carbon particle are being re-worked throughout the underlying sediment column. The width of each image is 14.5 cm (images cleared for public release).

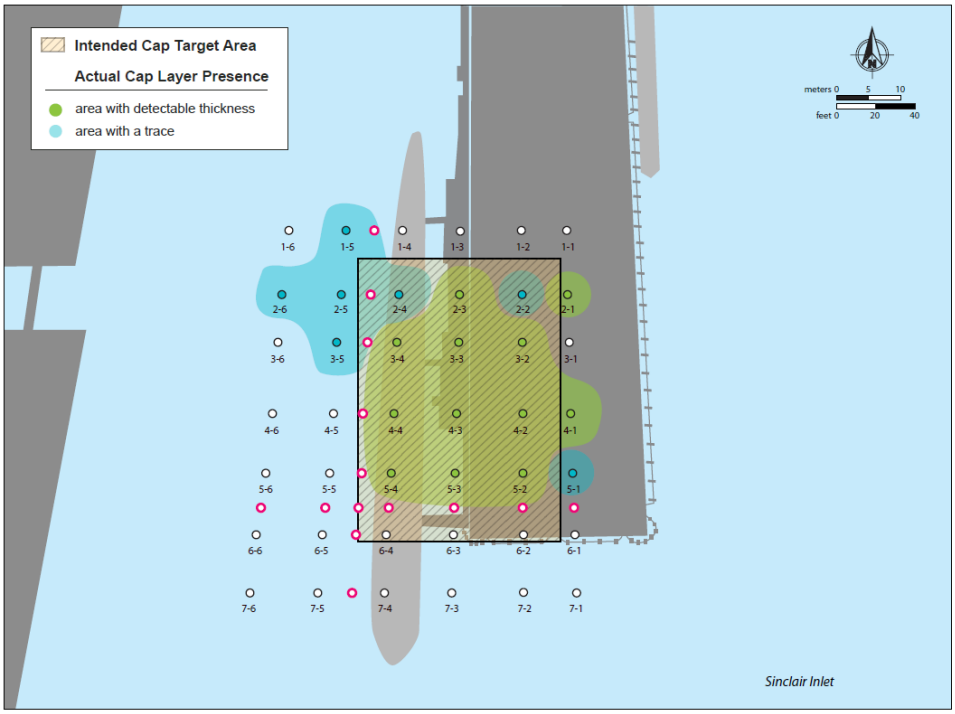


Figure 11. Comparison of intended cap target area with the actual cap presence measured in the October 2012 SPI survey.

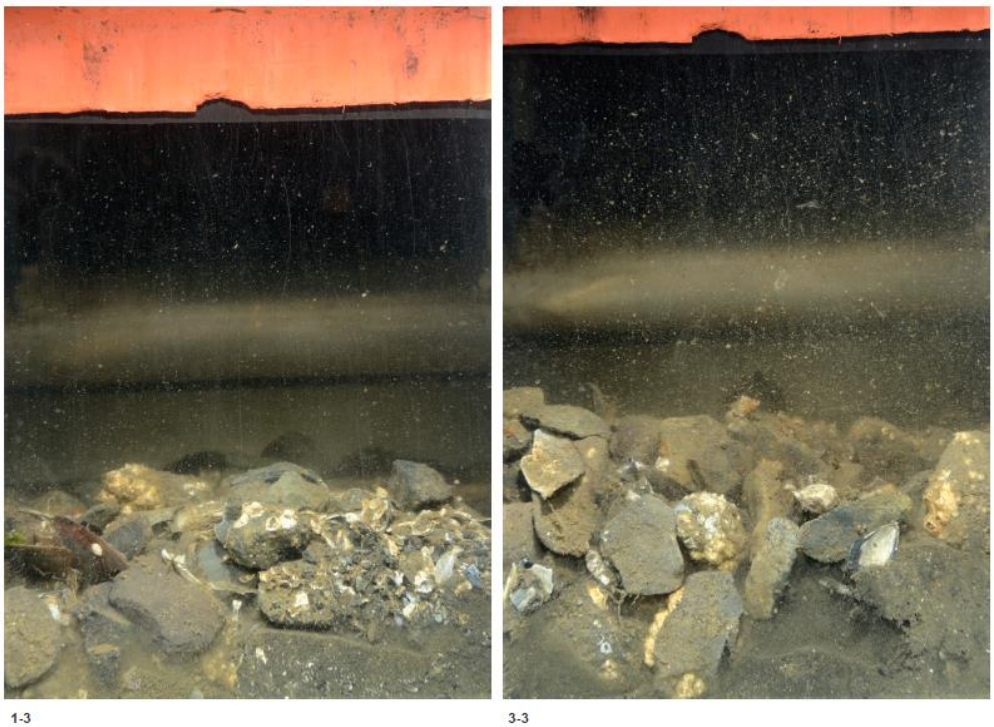


Figure 12. Sediment profile images from SPI stations 1-3 (left) and 3-3 (right) showing shells and cobbles over finer sediments. The cobbles are likely the remnant of the "sand blanket" placed along the pier following fender pile replacement in 2010. The width of each profile image is 14.6 cm (images cleared for public release).

Figure 13. Comparison of intended cap target area with the actual cap presence measured in the August 2013 (10-month) SPI survey.

Results of the 0.5 (October 2012) and 10-month (August 2013) SPI surveys indicated that the aggregate did not erode or mound up in areas. Diver video during August 2013 survey also confirmed this, as well as amendment presence on the steep side-slopes at the base of the pier. At the SPI station nearest to station 2, only a trace amount of amendment was observed in the 3-month and 10-month SPI investigations, and no aggregate was observed in cores obtained during the 10-month investigation. Because this area/station was not amended sufficiently, it was not included in the comparison of remedial performance (differences in carbon content) or effectiveness (differences in PCB availability).

Figure 14: This profile image from SPI station 4-4 shows active particle transport of the activated carbon particles as well as development of a surface oxidized layer. The width of profile image is 14.6 cm (images cleared for public release).

Sediment Cores: Following placement significant increases in the TOC content in the surface sediments were observed. For the 5-10 cm layer, it appears to have needed more time to work down, since only the 10-month data indicate a significant difference from the control (**Figure 15a**). For the 10-15 cm layer, it looks like it may be increasing slightly (although not significant). The black carbon in surface layer remained relatively constant, while there was a marked increase in black carbon in the 5-10 cm and 10-15 cm layers after 10 months (**Figure 15b**). In general, the measurements of TOC confirm an increase in carbon content in sediment. There is an approximate increase in surficial (0-5 cm) layers from 4% to 8% TOC. However, TOC and BC concentrations were highly variable among stations, there was a lot of heterogeneity within the stations, and sample processing was complicated by widely varying amounts of shell hash, cobble, and aggregate that could have biased the results obtained.

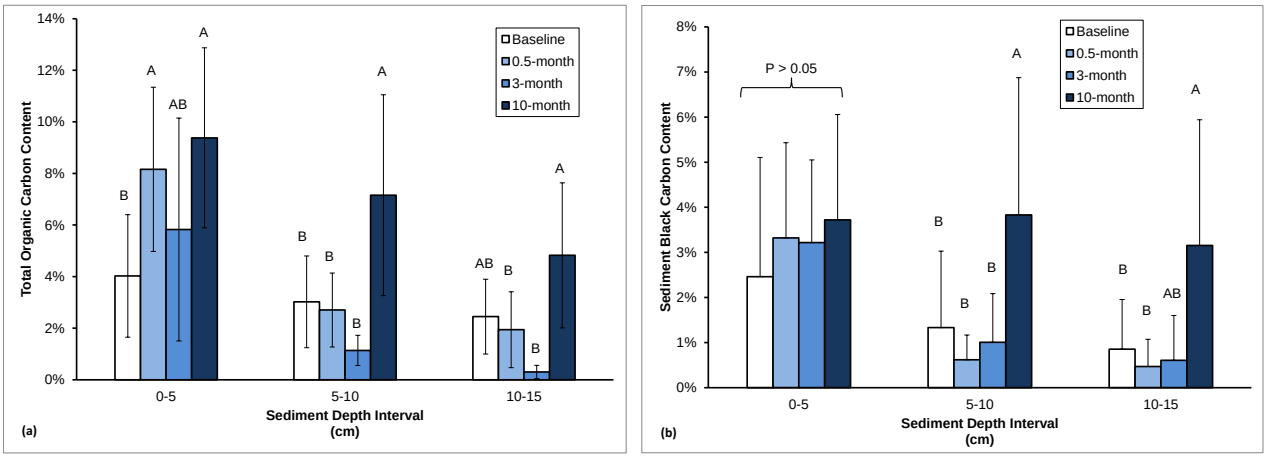


Figure 15. Total organic carbon (TOC, a) and black carbon (BC, b) content in sediment core samples at three depth intervals.

Short-term Performance Assessment

The results for PCB concentrations in benthic invertebrates, porewater and sediment from the baseline and T=10-month investigations were used to evaluate the short-term effectiveness of the amendment in reducing contaminant bioavailability.

PCBs in Benthic Invertebrates

The concentration of total PCBs in both clams and worms decreased approximately 80% following placement of the activated carbon amendment, indicating success in reducing PCB bioavailability at the 9 stations that received amendment. The concentration of total PCBs in *M. nasuta* decreased on average by 74% (ninety-five percent confidence interval [CI] 48-87%), from an average baseline value of 930 ng total PCBs/g, lw to 250 ng total PCBs/g, lw (p = 0.06, **Figure 16a**). The concentration of total PCBs in *N. caecoides* decreased on average by 87% (CI 66-95%), from an average 2,100 ng total PCBs/g, lw to 280 ng total PCBs/g, lw (p = 0.04, **Figure 16b**).

There were several suspect data points in the highly variable tissue PCB data. Upon further inspection, two extremely low values were noted for *M. nasuta* at station 4 in the baseline investigation (46 ng total PCBs/g, lw) and at station 9 in the 10-month investigation (150 ng total PCBs/g, lw). After exclusion of these potential outliers, a stronger statistical difference between the baseline and 10-month concentrations was observed (*P* = 0.03), confirming the difference in baseline and 10-month results, and indicating an average decrease in concentrations of PCBs of 78% (CI 58-88%). Additionally, an extremely high suspect value (20,000 ng/g, lw) was noted for *N. caecoides* at station 10 in the 10-month investigation. This value was the second highest concentration measured for all the tissues in both investigations, and was considered to be an outlier. When the potential outlier was removed, the statistical significant difference between the baseline and 10-month concentrations was maintained, however the average (95% CI) decrease in concentrations from the baseline changed from 87% % (66-95%) to 93% (91-95%), suggesting a larger decrease in total PCB concentrations.

Statistically significant decreases were also observed for concentrations of total tetrachlorinated, pentachlorinated, and hexachlorinated congeners in the two species (**Figure 16**). These homologue groups represented, 96% and 79% of the total detected PCBs in *M. nasuta* and *N. caecoides*, respectively. For the concentration of total pentachlorinated congeners in *N. caecoides*, the difference between baseline and 10-month (P = 0.08); however, the results were influenced by an extremely high outlier at station 10 in the 10-month investigation (12,000 ng total pentachlorinated congeners/g, lw). Although inspection of cores indicated the presence of aggregate at this station, results suggest only a trace amount of activated carbon may have been present in the sample, as this station was at the margin of the amendment area (**Figures 11 and 13**). The concentration of total pentachlorinated PCBs in *M. nasuta* at this station were 25% less than baseline 10-months following the amendment application, suggesting that amendment amounts may not have been sufficient at station 10. Removal of the potential 12,000 ng total pentachlorinated congeners/g, lw outlier resulted in detection of statistical significance (P = 0.008) in total pentachlorinated congeners in *N. caecoides* between the baseline and 10-month investigation. Total tetrachlorinated congeners in *M. nasuta* were marginally significant (P = 0.09), but when the two lowest results from the baseline and 10-month datasets were removed (as in the analysis of total PCBs in *M. nasuta*, described above), the difference was statistically significant (P = 0.04). All other differences noted in **Figure 16** were detectable at the α level of 0.05.

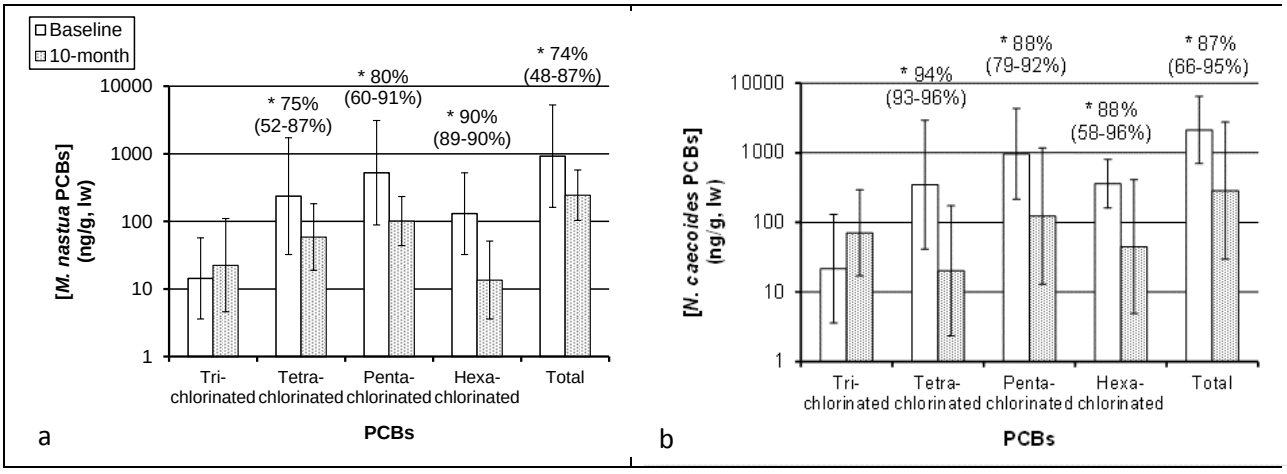


Figure 16. Mean (SD) concentrations of PCBs in *Macoma nasuta* (a) and *Nephtys caecoides* (b) measured during the pre-remedy (baseline) and post-remedy (10-month) investigations. Labels above the column pairs indicate average (95% CI) percent decrease observed between the baseline and 10-month results when a statistically significant difference (α = 0.05) was detected.

Trichlorinated congeners comprised the remainder of the detectable PCBs in the organisms, albeit at low concentrations (14 to 70 ng/g, lw) that were not statistically significantly different between the baseline and 10-month investigations (**Figure 16**). Trichlorinated PCB congeners are at least an order of magnitude less hydrophobic than the penta- and hexachlorinated congeners that comprised the majority of the bioaccumulated PCB, and it is possible that activated carbon may be less effective in absorbing congeners from this homologue group. However, decreases bioaccumulation were evident among the tetra-, penta-, and hexachlorinated congeners and the differences tended to increase with level of chlorination (**Figure 16**) Other studies indicated that activated carbon is effective in reducing

bioavailablity of of trichlorinated PCB congeners (Zimmerman et al., 2004). The accumulated trichlorinated PCBs may reflect accumulation of trichlorinated PCBs from surface water due to partial exposure of organisms to the sediment-water interface; this exposure pathway may not be influenced greatly by activated carbon in the sediment. More work in confirming bioavailability reductions with less hydrophobic compounds (such as trichlorinated PCBs) as a result of activated carbon amendment would be helpful.

PCBs in Sediment Porewater

In concurrence with the bioaccumulation results, the concentration of freely-dissolved total PCBs in surface sediment also decreased greatly following amendment with the activated carbon amendment (**Figure 17**). Total PCBs (tetra-, penta-, and hexachlorinated biphenyls) in sediment porewater at the 9 stations that received amendment showed a statistically significantly decreased ($P < 0.0001$) by 90% (CI of 81-95%) as a result of the amendment, with concentrations decreasing from 0.165 to 0.017 ng total PCBs/L (**Figure 17**).

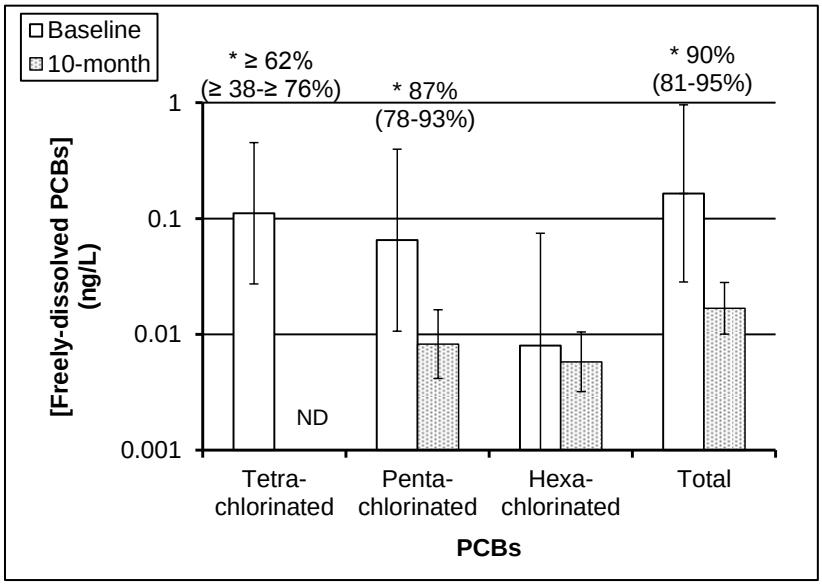


Figure 17. Mean (SD) concentrations of freely-dissolved PCBs measured with SPME passive samplers during the pre-remedy (baseline) and post-remedy (10-month) investigations. Labels above the column pairs indicate average (95% CI) percent difference observed between the baseline and 10-month results when a statistically significant difference ($\alpha = 0.05$) was detected. Tetrachlorinated congeners were not detected (ND) above concentrations of 0.03 to 0.13 ng/L during the 10-month investigation.

Significant decreases were observed for tetrachlorinated and pentachlorinated congeners, which comprised 71-88% of the total detected PCBs in the investigations. Concentrations of tetrachlorinated congeners were not detected in any of the samples in the 10-month investigation above detection limits ranging from 0.03 to 0.13 ng total PCBs/L. Using a semi-quantitative approach, we substituted detection limit values for the non-detect results in the 10-month tetrachlorinated congener dataset. A comparison of baseline and 10-month results indicates that at concentrations of freely-dissolved

tetrachlorinated congeners decreased significantly (P = 0.019) by an average (SD range) of at least 62% (38-76%). For the pentachlorinated congeners, mean (95% CI) concentrations decreased significantly (P < 0.0001) by 87% (78-93%).

In contrast, hexachlorinated congeners were detected at much lower concentrations (0.008 and 0.006 ng/L average for baseline and 10-month, respectively), and there was no evidence of a statistically-significant difference between the baseline and 10-month results. Only 24% of the samples yielded detectable results, so it is difficult to evaluate the efficacy of the remedy for this homologue group with high confidence. A decrease in the bioavailability of hexachlorinated PCBs was observed in the bioaccumulation results (**Figure 16**), so it is possible that increasing the sensitivity of the method would also indicate a difference in concentrations of freely-dissolved hexachlorinated PCBs.

PCBs in Sediment

As observed in the lab treatability study, PCB sediment concentrations decreased as well (**Figure 18**). While the exact nature of this decrease is not fully understood at this time, along with the observations from the treatability study, there is evidence in the literature (see Kupryianchyk et al. 2013) that concentrations of both PCBs and PAHs are lower in sediments treated with powdered activated carbon as compared to the unamended sediments. This could be explained by a decrease in the ability to extract the PCBs from the sediments treated with the activated carbon due to binding of the PCBS to the carbon particles. It is also possible that some portion of the decrease in the total PCB sediment concentrations could be a result of a mixture of newly deposited cleaner sediment and addition of the non-aggregate fraction of the amendment creating a less contaminated surface over time.

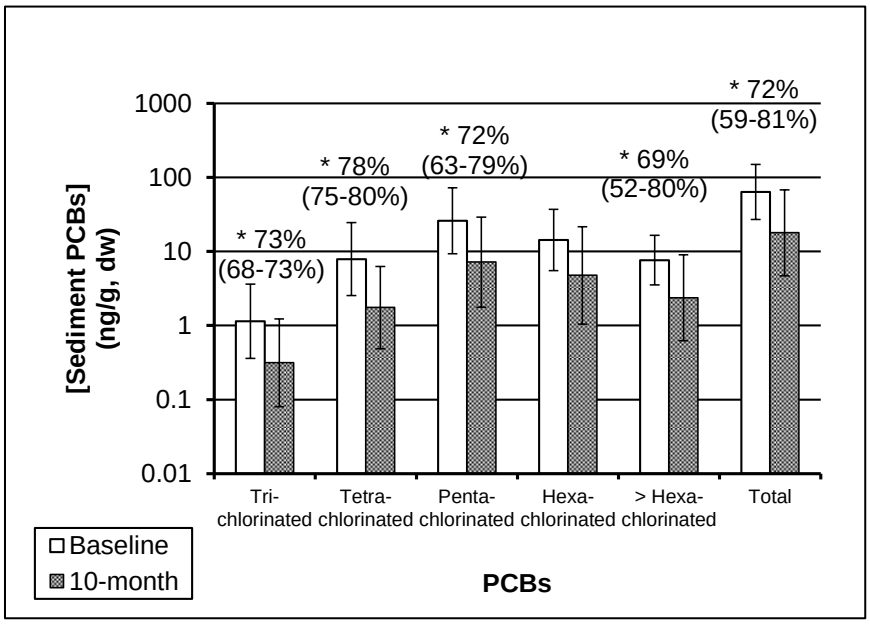


Figure 18. Mean (SD) concentrations of PCBs in surface sediment measured during the pre-remedy (baseline) and post-remedy (10-month) investigations. Labels above the column pairs indicate average (95% CI) percent decrease observed between the baseline and 10-month results when a statistically significant difference (α = 0.05) was detected.

Benthic Community Health

Benthic Community Census

Benthic community monitoring for the baseline and 10-month post amendment deployment sampling events indicated abundance at the amended stations decreased between baseline and 10-month (post-amendment) surveys, but was driven by nematode abundance decreases. Compared to the baseline, average total abundance decreased significantly (P = 0.03) by nearly 60% in stations where the amendment was applied, but remained consistent in reference stations (P = 0.67) (**Figure 19**). The decrease in nematode abundance from the baseline to the 10-month event represented a significant (P = 0.002) decrease of 85% (**Figure 20**). Abundance of non-nematode invertebrates at the amended stations was comparable to that of the reference stations. There was no significant difference (P = 0.69) between average baseline (6,900 individuals/m^2) and 10-month (8,800 individuals/m^2) abundance of non-nematodes at the amended stations. Also, there was no significant difference (P = 0.88) between average baseline (2,400 individuals/m^2) and 10-month (2,500 individuals/m^2) abundance of non-nematodes in the reference stations (**Figure 21**).

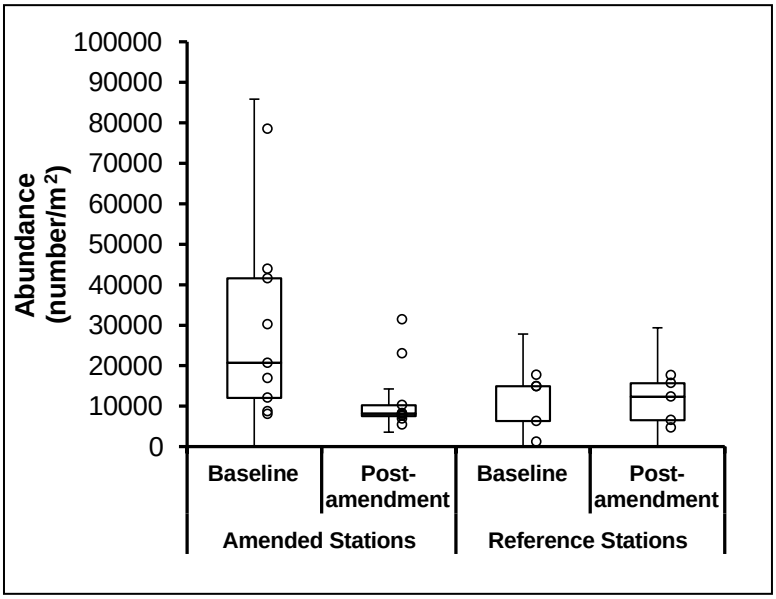


Figure 19. Total abundance at amended and reference stations for the baseline and post-amendment placement (T=10 month) surveys.

The decrease in nematode abundance allowed other characteristics of the benthic community health to increase. Four benthic community metrics related to diversity (diversity, evenness, dominance, and abundance of the most dominant taxa) indicated significant positive effects of the amendment on benthic community health. The amendment appears to have reduced the dominance of nematodes on the benthic community, promoting the diversity of other taxa. An adverse effect of the amendment on annelid abundance (considered to be at risk due to effects of carbon ingestion) was not observed at the amended stations. Overall, although the abundance of nematodes decreased at stations within the remedy footprint, the composition of the community became more diverse and evenly abundant as a

result of the amendment addition. As a result, the effect of the amendment on overall benthic community health is considered neutral.

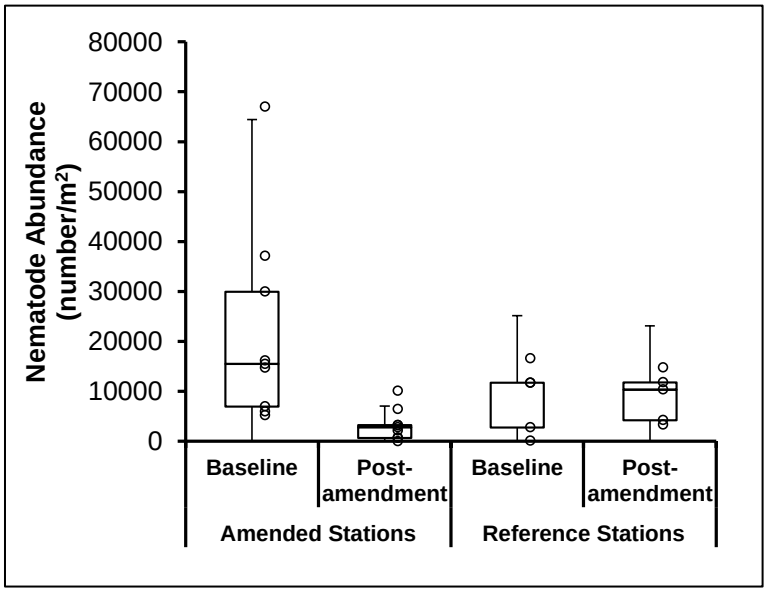


Figure 20. Total nematode abundance at amended and reference stations for the baseline and post-amendment placement (T=10 month) surveys.

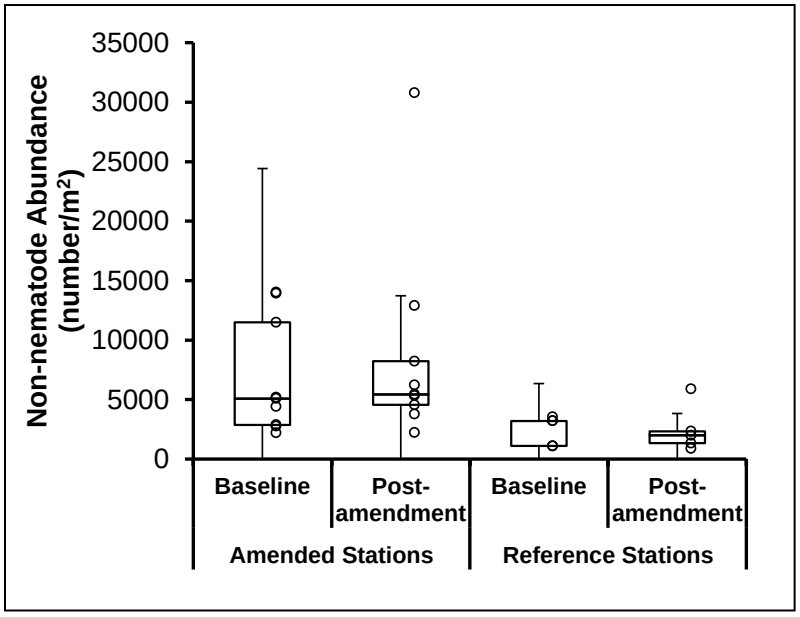


Figure 21. Total non-nematode abundance at amended and reference stations for the baseline and post-amendment placement (T=10 month) surveys.

SPI Camera

The mapped distribution of infaunal successional stages for the baseline, T=0.5 month and T=10 month surveys is shown in **Figure 22**. Results from the 2012 post-placement survey (T=0.5 months) indicated that while there was a noticeable change in biological community status compared to baseline conditions because of the recent disturbance to the area from the cap placement, presence of Stage 3 taxa (infaunal deposit feeders) was evident at 19 of the 42 stations. Three of the stations outboard of the pier (Stations 3-4, 4-4, and 5-4) that formerly had dense assemblages of tubes from large sabellid polychaetes in the baseline survey were devoid of any of these assemblages after cap placement (**Figure 23**). Ten months after placement (2013) there was a noticeable improvement in biological community status under the pier compared to the post capping survey done in 2012. However, there was a retrograde in successional status at some of the stations outboard of the pier (Stations 1-5, 1-6, 2-4, 3-4, and 3-6). The presence of Stage 3 taxa (larger infaunal deposit feeders) was evident at 18 of the 50 stations (Germano & Associates, 2013; 2014). The results from the 2013 survey showed that the active cap amendment is being reworked into the sediment by the resident infauna (**Figure 14**). Over time, the reappearance of Stage 3 taxa as well as thicker oxidized layers should be more widespread, depending on the frequency of physical disturbance to the bottom from propwash and ship activity.

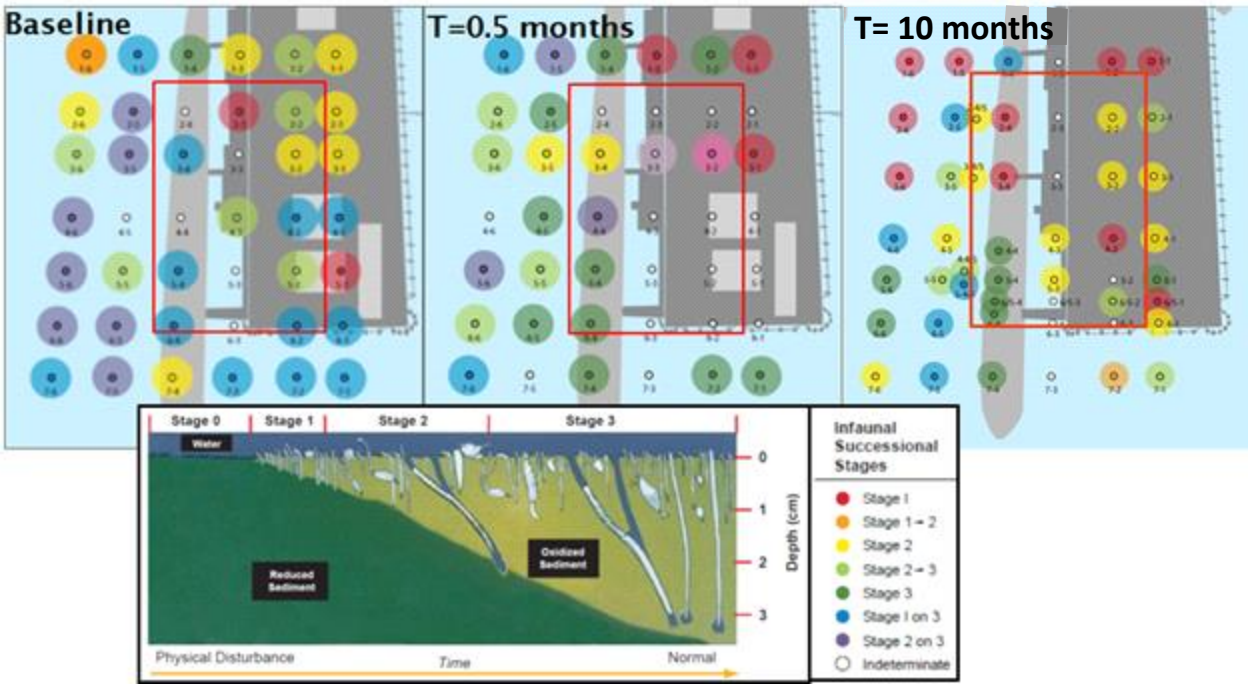


Figure 22. Infaunal successional benthic stages for Baseline, T=0.5 months and T=10 months as determined from SPI.

Figure 23. These profile images from Station 5-4 taken before (left) and after (right) cap placement show how placement of the cap material eliminated the assemblage of large sebellid polycheate tubes that were present during the baseline survey. Scale: width of each image = 14.5 cm (images cleared for public release).

SUMMARY

The results from this study, to date, have evaluated the placement and short-term stability of the amendment, the short-term effectiveness of the amendment in controlling contaminant bioavailability, and evaluated the initial response of the benthic community to the application of the amendment over the target area.

From an engineering perspective, the placement of the amendment over the 0.5 acre target area, which extended from the berthing area underneath the pier, was successful. Sediment profile imaging, sediment core samples and diver surveys performed two weeks after placement of the amendment material showed that 90% of core target area received the amendment and 70% of the target area received target thickness (5 cm) or more. The amendment was still found to be present ten months after placement although there were some locations where the layer that was obvious in 2012 was no longer visible in 2013, and conversely where there was no detectable layer in 2012 appeared to be present in 2013. The long-term stability of the amendment will continue to be monitored through two more surveys (214 and 2015).

In terms of reduction in contaminant bioavailability, the concentration of total PCBs in both bioaccumulation test organisms decreased approximately 80% as a result of the activated carbon amendment, indicating success in reducing PCB bioavailability at the 9 stations that received amendment. In concurrence with the bioaccumulation results, the concentration of concentration of total freely-dissolved PCBs in surface sediment also decreased greatly as a result of the activated carbon amendment. Total PCBs (tetra-, penta-, and hexachlorinated biphenyls) in sediment porewater at the 9 stations that received amendment significantly decreased ($P < 0.0001$) by 90% (95% CI of 81-95%) as a result of the amendment, with concentrations decreasing from 0.165 to 0.017 ng total PCBs/L.

In terms of overall impact to the benthic community, compared to the baseline, the average total abundance decreased significantly (P = 0.03) by nearly 60% in stations where the amendment was applied, but remained consistent in reference stations (P = 0.67). The decrease in nematode abundance from the baseline to the 10-month event represented a significant (P = 0.002) decrease of 85%; however the abundance of non-nematode invertebrates at the amended stations was comparable to that of the reference stations. There was no significant difference (P = 0.69) between average baseline (6,900 individuals/m^2) and 10-month (8,800 individuals/m^2) abundance of non-nematodes at the amended stations. The decrease in nematode abundance allowed other characteristics of the benthic community health to increase.

The overall results suggest that the short-term objectives of this project have been demonstrated to be successful. The amendment was placed over the majority of the area at the desired thickness (~2 inches) and is being reworked into the sediments at the site. Bioavailability of the PCBs to the benthic organisms has been drastically reduced in response to the addition of the activated carbon amendment. Finally, while there was an impact to the nematode population immediately following the addition of the amendment, the addition of amendment also appears to have allowed for more diversity among the other organisms.

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