



Research article

Polychlorinated biphenyl (PCB) contamination of subsistence species on Unalaska Island in the Aleutian Archipelago



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ABSTRACT

Polychlorinated biphenyls (PCBs) are a group of synthetic, lipophilic organochlorines that were banned due to their impacts on human and wildlife health and environmental persistence. Although banned, the continued release from pre-banned products allows them to persist at toxic levels in the environment. This is especially the case in lipid rich food webs of the Arctic, where PCBs accumulate due to both long-range atmospheric transport and locally contaminated sites such as formerly used defense (FUD) sites. At the request of the leadership of the Qawalangin Tribe of Unalaska Island in the Aleutian Archipelago, we analyzed PCB concentrations in samples of threespine stickleback (*Gasterosteus aculeatus*) and subsistence foods (i.e., salmonid species and blue mussels [*Mytilus edulis*]) collected at both FUD and non-FUD sites. PCBs were extracted from samples using a QuEChERS method. The mean PCB concentrations across all mussel samples was 6.1 ppb; mussels from FUD sites had nearly double the PCB concentrations (7.6 ppb) compared to non-military sites (3.9 ppb), and at two FUD sites the PCB concentrations exceeded safe consumption guidelines. The mean total PCB concentration for fish was 2.8 ppb; fish PCB concentrations were higher at FUD sites (3.2 ppb) compared to non-military sites (1.2 ppb). These results support the need to remediate the FUD sites of “Building 551/T Dock to Airport” and “Delta Western”. More generally, these results provide further evidence of the continued problem of PCB contamination at FUD sites in the Arctic, many of which are co-located with indigenous communities.

1. Introduction

In the United States the commercial production of PCBs began in 1929 and continued until the late 1970's when it was determined that bioaccumulation, environmental persistence, and long-range transport accompany their release, making them a persistent organic pollutant (POP) (Garmash et al., 2013; Gioia et al., 2013). Due to their chemical and thermal stability, the primary use of PCBs was in dielectric fluid in transformers and capacitors (Hardell et al., 2010). Plasticizers, lubricating oils, hydraulic fluid, paint, and ink were other common applications of PCBs (Carpenter, 2006; Gioia et al., 2013; Hardell et al., 2010). Although banned in the United States since 1979 (EPA, 1979) and internationally since 2001 (Porta and Zumeta, 2002; UNEP, 2001), the continued release of PCBs via both point and diffuse sources (e.g., landfills, transformers and capacitors, volatilization of previously released PCBs, abandoned military installations, byproducts of waste

incineration) remains an issue of concern for both human and wildlife health (Blais, 2005; Garmash et al., 2013; Gioia et al., 2013).

The differing number and placement of chlorine atoms on the biphenyl ring results in 209 possible PCB congeners with unique physical and biological properties (Hardell et al., 2010). Relatively light molecular weight PCBs undergo long-range transport via global distillation and accumulate at high latitudes due to year-round low temperatures (Bard, 1999; Muir et al., 1999; Wania and Mackay, 1993). Furthermore, the low temperatures and low intensity sunlight of the Arctic reduce the rate of degradation of PCBs (Garmash et al., 2013; Wania and Mackay, 1993).

PCBs are among a group of hydrophobic contaminants that are not easily metabolized, causing them to accumulate within the adipose tissues of organisms (Muir et al., 1999), including many long-lived fish and marine mammals (Ayotte et al., 1995; Bard, 1999; Wania and Mackay, 1993). The primary route of PCB exposure for humans and animals is consumption of contaminated food (Carpenter, 2006). Once incorporated into the food web, PCBs bioaccumulate and biomagnify, especially

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in the lipid rich tissue of arctic animals (Bard, 1999; Hardell et al., 2010). High trophic level marine animals are at risk for exposure to high levels of PCBs, as are the subsistence communities with high levels of consumption of these animals (Ayotte et al., 1995; Wania and Mackay, 1993).

Due to the variation in physical properties, different PCB congeners cause a variety of health effects including alteration or suppression of immune, thyroid and reproductive functions as well as reduced cognitive function and increased risk of developing cancer (Carpenter, 2006; Hardell et al., 2010; Schell et al., 2008). The most notable health risks associated with consumption of PCBs are effects on reproduction and development (Ayotte et al., 1995). PCBs cross the placental barrier and accumulate in the developing fetus as well as in breast milk (Bard, 1999; Dewailly et al., 1993). Reduced intelligence and cognitive function resulting from exposure to PCBs cannot be reversed (Carpenter, 2006). These health consequences of PCB exposure are particularly relevant to arctic indigenous communities subsisting on high trophic level foods (Ayotte et al., 1995; Hardell et al., 2010).

The Aleutian Islands of Alaska are included in the circumarctic region within the arctic geographical coverage of the Arctic Monitoring and Assessment Programme (AMAP), placing the Aleutian Archipelago within the area of concern for arctic contamination. The Aleutian Archipelago contains over 300 volcanic islands that extend 1600 km west from the Alaska Peninsula in the northern Pacific Ocean. The Aleutian Islands are important habitat and breeding grounds for ten million seabirds (Byrd et al., 2005).

PCB contamination in arctic areas with a history of military use can stem from atmospheric deposition and point sources on military sites, especially formerly used defense (FUD) sites. Due to their proximity to East Asia, the western-most islands of the Aleutians (the Near Islands) were invaded by the Japanese during World War II, which led to the militarization of much of the Aleutian Archipelago by U.S. forces. Subsequent to World War II, their proximity to the Soviet Union led to the creation of Cold War military installations. Alaska contains approximately 600 FUD sites (Scrudato et al., 2012; von Hippel et al., 2016), many of which, including many in the Aleutians, are contaminated with PCBs (Hardell et al., 2010). Many FUD sites are located in or near indigenous villages or on important subsistence land (Scrudato et al., 2012; von Hippel et al., 2018; von Hippel et al., 2016).

The island of Unalaska within the Aleutian Archipelago (Figure 1) contains the largest town in the Aleutians, the city of Unalaska, with a

population of 5000 residents that increases during the summer commercial fishing season (USCB, 2018). Unalaska also contains approximately 30 FUD sites (Blais, 2005; Scrudato et al., 2012). Dutch Harbor on the island of Unalaska is a commercial fishing area of global significance that is also the basis of the local economy. Fish and other organisms harvested from this area are consumed by both the Qawalangin Tribe of Unalaska, who rely on subsistence animals, and recipient populations. Subsistence activities are important both culturally and for nutrition, but may also lead to ingestion of unsafe levels of POPs such as PCBs (Ayotte et al., 1995; Muir et al., 1999).

Due to the remote locations of many Alaskan FUD sites, and the small populations that often live near such sites, remediation standards are often less strict than in more urban settings (Scrudato et al., 2012). The FUD sites pose potential health risks for Unalaska residents, who did not elect to host military installations and yet may experience adverse effects from military contaminants. Hence, at the request of the leadership of the Qawalangin Tribe, we quantified the PCB concentrations of select subsistence species in both marine (blue mussels [*Mytilus edulis*]) and freshwater habitat (salmonid species) at both FUD and non-military sites. In addition to subsistence species, we sampled threespine stickleback (*Gasterosteus aculeatus*) in fresh water because of their importance as a monitoring species in environmental toxicology (von Hippel et al., 2016).

2. Materials and methods

2.1. Specimen collection and storage

Blue mussels, threespine stickleback, and juveniles of various salmonid species were collected from June 10–26, 2017 from both freshwater and marine sites on the island of Unalaska (Table 1, Figure 2). On each collection day, specimens were collected from multiple sites. During low tide, mussels were collected by hand from intertidal zones. Mussels were collected from different substrates and positions within each site to be representative of sites of harvest by the local community and to capture any potential variation in PCB levels by position within the intertidal zone. Mussels were selected based on size, with preference given to larger individuals, and those without broken shells. Two mussels were analyzed from each site with the exception of Wislow and Cascade Falls for which only one composite mussel sample was analyzed per site, due to small mussel size at these locations. Salmonids and stickleback were sampled from freshwater locations (n = 10 for each fish family at

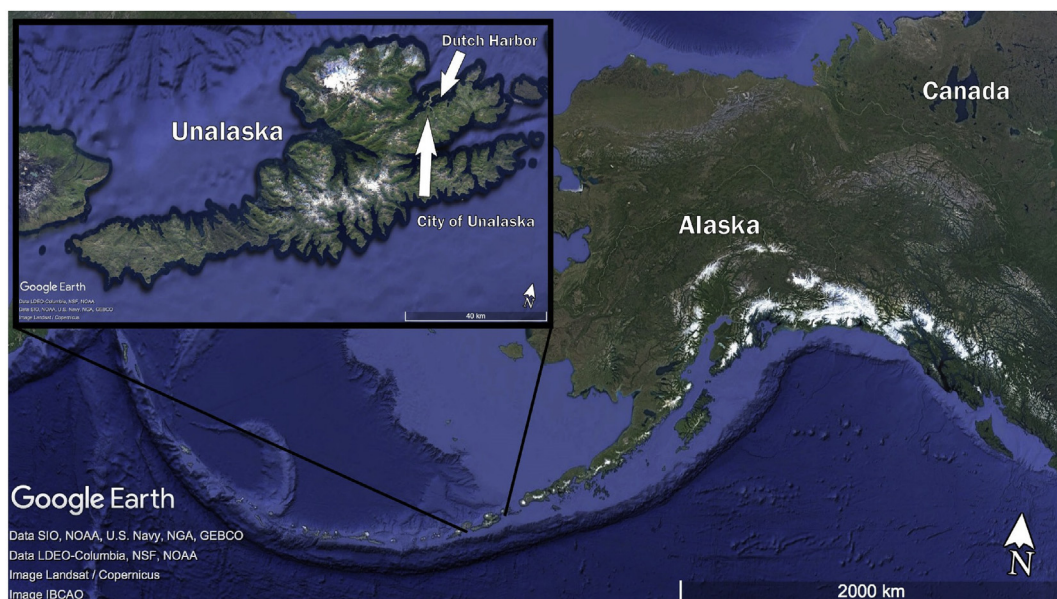


Figure 1. Location of the island of Unalaska in the Aleutian Archipelago. Image derived from Google Earth.

Table 1. Sampling sites on Unalaska.

Site Name	Formerly Used Defense Site?	Species Collected	Latitude (°N)	Longitude (°W)
APL/Rocky Point	Military	Blue Mussel	53.888171	166.526738
Captains Bay - Rt side (Crowley) Dock	Military	Blue Mussel	53.852301	166.569865
Captains Bay - Lf side (Crowley) Dock	Military	Blue Mussel	53.847266	166.578966
Captains Bay - Port Levashef	Military	Blue Mussel	53.83701	166.61732
Delta Western	Military	Blue Mussel	53.889898	166.53374
Front Beach	Military	Blue Mussel	53.885466	166.555203
Inside Dutch Harbor Spit	Military	Blue Mussel	53.904176	166.511461
Margarets Bay Entrance	Military	Blue Mussel	53.880543	166.548745
Royal Aleutian Dock #1	Military	Blue Mussel	53.881965	166.544153
Royal Aleutian Dock #2	Military	Blue Mussel	53.88165	166.541325
S Curves	Military	Blue Mussel	53.87753	166.565441
Sub Base	Military	Blue Mussel	53.878035	166.552895
Sub Base (Waleshek) Dock	Military	Blue Mussel	53.877883	166.555591
Sub Base Entrance	Military	Blue Mussel	53.876236	166.549375
Sub Base Haul Out	Military	Blue Mussel	53.877901	166.556416
Building 551/T Dock to Airport	Military	Blue Mussel	53.893015	166.536995
Unalaska Dump	Military	Blue Mussel	53.881233	166.511613
Wide Bay	Military	Blue Mussel	53.95234	166.62553
Capt Bay - Fox Site	Non-Military	Blue Mussel	53.867926	166.546333
Capt Bay - Left side Cannery	Non-Military	Blue Mussel	53.85714	166.556358
Capt Bay - Rt side Cannery	Non-Military	Blue Mussel	53.858605	166.55186
Cascade Falls	Non-Military	Blue Mussel	53.93166	166.64273
Constantine Bay	Non-Military	Blue Mussel	53.95692	166.409855
Rat Islands	Non-Military	Blue Mussel	53.84848	166.50273
Wislow	Non-Military	Blue Mussel	53.99942	166.726433
Morris Cove Creek	Military	Salmonids and Threespine Stickleback	53.916703	166.429546
Unalaska Lake	Military	Salmonids and Threespine Stickleback	53.86418	166.520698
Matson Lake	Military	Threespine Stickleback	53.887175	166.540236
Nateekin River	Non-Military	Salmonids and Threespine Stickleback	53.8718	166.63892
Shaishnikoff River	Non-Military	Salmonids	53.824665	166.610461

each site found except Nateekin River which had $n = 3$ for each due to low sampling success; Table 1). Fish were collected using unbaited 0.64 cm wire-mesh minnow traps. Fish were euthanized with an overdose of pH neutral MS-222 anesthetic.

Samples were labeled and held inside a cooler in the field for approximately 1 h and then transferred to a -20 °C freezer. At the completion of fieldwork, the samples were shipped frozen overnight to the lab at Northern Arizona University and stored at -80 °C until PCB extraction and analysis. All collection protocols were approved by the Alaska Department of Fish and Game (collection permit SF2017-127) and all animal protocols/ethics were approved by the Northern Arizona University Institutional Animal Care and Use Committee (IACUC protocol 17-003).

2.2. QuEChERS PCB extraction

PCB extraction was conducted on whole body homogenate (fish) or soft tissue homogenate (mussels) using the “modified QuEChERS (quick, easy, cheap, effective, rugged, safe) method” described by Chamkasem et al. (2016). The solvent to sample ratio used in this method increases the efficiency of PCB congener extraction as compared to previous methods. Using this QuEChERS extraction approach, recovery of PCB congeners is above 70%, and for each congener the recovery rate is consistent (average RSD = 5.1%) over a wide range of PCB concentrations tested from 10 to 300 ng/g (Chamkasem et al., 2016). Thus, the extraction approach and analytical procedures likely resulted in an underestimation of PCB concentrations in the samples described here. Samples were removed from -80 °C, kept on ice and allowed to thaw. Using a knife and dissection scissors, sample tissue was thoroughly

homogenized. Dissection tools were sterilized between each sample using Alconox Powdered Precision Cleaner (White Plains, NY, USA). All fish were processed through additional homogenization using the Fisher Scientific PowerGen 1000 S1 homogenizer (Pittsburgh, PA, USA) and Fisher Scientific FB 505 sonicator (Pittsburgh, PA, USA) to optimize available surface area for PCB extraction.

A homogenate mass of 3 g wet weight was placed into a 50 mL centrifuge tube. If the mass of an individual specimen did not meet the 3 g requirement, multiple individuals from the same site were combined to make a single sample. All stickleback from Morris Cove Creek, Unalaska Lake, Matson Lake, and Nateekin River were composite samples as well as two salmonid samples from Nateekin River and one from Morris Cove Creek. Salmonid samples were not identified to the genus and species level. Deionized water (5 mL) and acetonitrile (30 mL) were added to the sample tubes. Tubes were shaken on a Glas-Col large capacity mixer, speed set on 50 (Terre Haute, IN, USA) for 30 min. MgSO_4 (6 g) and NaCl (1.5 g) were added to the sample tubes, which were put on the shaker for another 10 min. Sample tubes were then centrifuged at 3000 rpm for 10 min (Sorvall Legened XTR, Osterode am Harz, Germany). The centrifuge speed (3000 rpm rather than 5000) is an exception to the method outlined in Chamkasem et al. (2016) due to the rotor capabilities of our centrifuge. Acetonitrile extract (1 mL) was pipetted into a 2 mL prepared QuEChERS centrifuge tube (unitedchem.com, Bristol, PA, USA) containing 150 mg of anhydrous MgSO_4 , 150 mg of PSA sorbent and 50 mg of CEC18 sorbent. The tubes were capped and placed on the shaker for 1 min. The tubes were then centrifuged at 2000 rpm for 10 min. The final sample extract (~500–600 μL) was transferred into a 1.5 mL glass vial. The final extracts were kept at -80 °C until shipment by overnight courier to the Arizona Laboratory for Emerging Contaminants (ALEC) in Tucson.

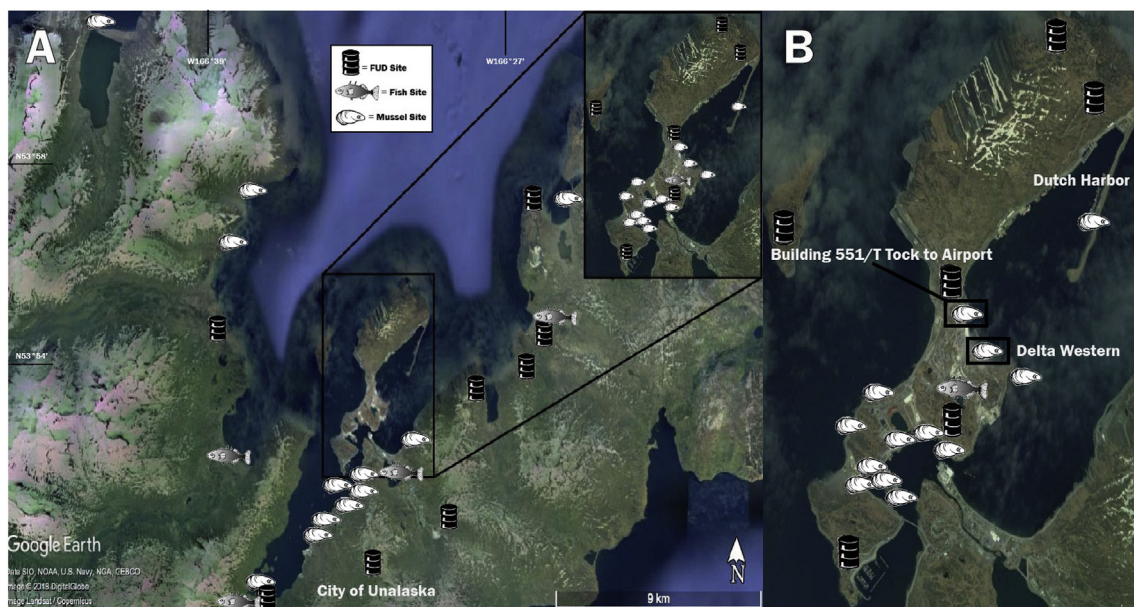


Figure 2. A: Sites sampled across Unalaska Island. Mussel and fish symbols indicate organism sampled at that location, and a barrel indicates a FUD site. B: Two sites with highest levels of PCB contamination, “Building 551/T Dock to Airport” and “Delta Western”. Mussels were sampled at both sites. Image derived from Google Earth.

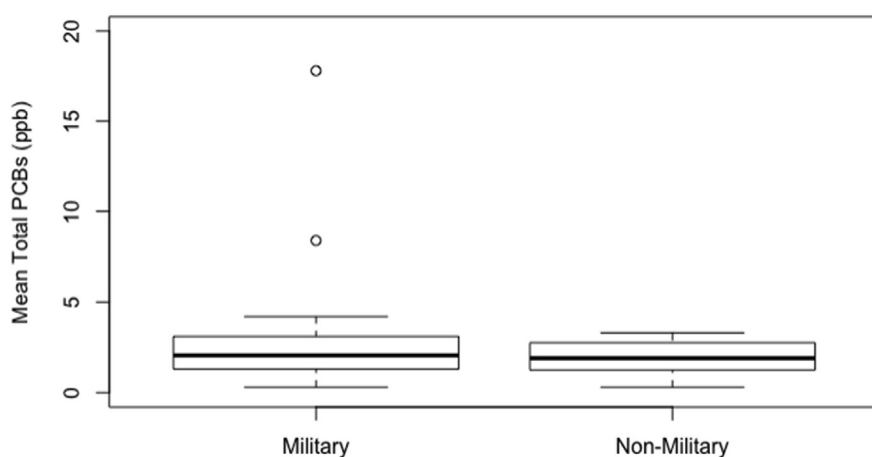


Figure 3. Mean PCB concentrations in parts per billion across all mussel sites sampled comparing military vs non-military sites. Bootstrapping for all mussel samples resulted in a mean total PCB concentration of 6.1 ppb. The analysis resulted in a mean total PCB concentration of 7.6 ppb for military sites, 3.9 ppb for non-military sites, and a difference between the two of 3.7 ppb. The PCB concentrations found in mussels were especially elevated at two Military/FUD sites; “Building 551/T Dock to Airport” and “Delta Western” showed mean total PCB concentrations of 81.4 ppb (data point not shown; see Figure 5) and 17.8 ppb, respectively.

PCBs concentrations in fish samples were quantified from five sites, while concentrations in mussel samples were quantified from 25 sites (Table 1). Twenty-one of these 30 sites were FUD sites (18 sampled for mussels, three for fish), while the other nine sites were non-military (seven mussel, two fish).

2.3. PCB analysis by GC-MS

Prepared sample extracts were analyzed using an Agilent GC (7890 gas chromatograph with 7683 autosampler; Santa Clara, CA, USA) using a split-splitless inlet in splitless mode. With direct autosampler injection of 1 μ l of sample the analytes were separated using an SPB-Octyl column (Supelco; 30 m $\times$ 0.25 mm $\times$ 0.25 μ m) using He as the carrier gas at a flow rate of 1.1 ml/min. The column temperature ran at 75 $^{\circ}$ C for 2 min, increasing 15 $^{\circ}$ C/min to 150 $^{\circ}$ C, then increasing 2.5 $^{\circ}$ C/min to 280 $^{\circ}$ C.

The GC was coupled with electron impact mass spectrometry using a Waters Quattro micro triple quadrupole mass spectrometer (Milford, MA, USA) in selected ion recording (SIR) mode. The ion transfer line temperature was 280 $^{\circ}$ C and the ion source was run at

220 $^{\circ}$ C with trap current at 200 μ A and electron energy of 70 eV. Presence and abundance of 23 PCB congeners was measured by comparison based on standard calibrant mixture C-SCA-06 (Accustandard Inc, New Haven, CT, USA). The standard mixture was diluted with nonane (Frontier Scientific, Logan, UT, USA) on an analytical balance to prepare the calibrants. Software used to acquire data from the GC-MS was MassLynx 4.1 and TargetLynx, respectively.

2.4. Statistical analysis

Statistical analyses were completed in R version 3.5.0. Resampling via bootstrapping was conducted to calculate the means and 95% confidence intervals for total PCB concentration contained in mussel and fish samples from each site. Additionally, bootstrapping was used to determine the detectable difference between the PCB concentration contained in mussel and fish samples at military vs. non-military sites. In order to facilitate the bootstrapping approach, the minimum detection limit was used as the data point for any congener that tested below the detection limit.

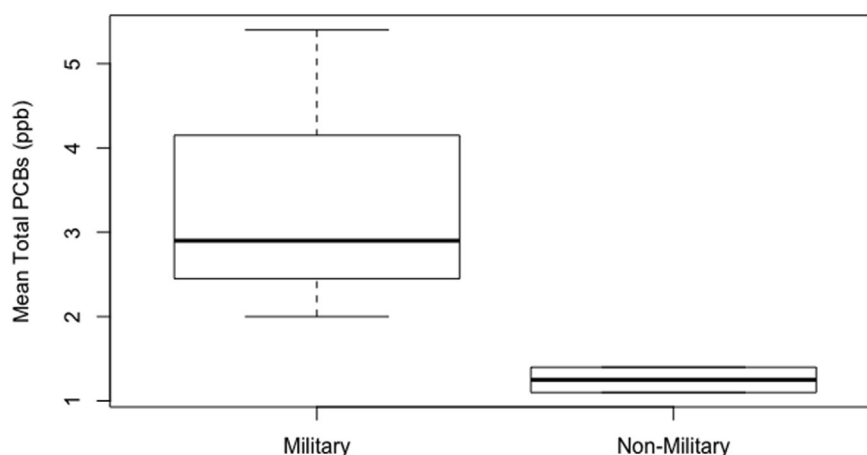


Figure 4. Mean PCB concentrations in parts per billion (ppb) across all fish sites sampled comparing military vs non-military sites. Bootstrapping for all fish samples resulted in a mean total PCB concentration of 2.8 ppb. The analysis resulted in a mean PCB concentration of 3.2 ppb for military sites, 1.2 ppb for non-military sites, and a difference between the two of 2.0 ppb.

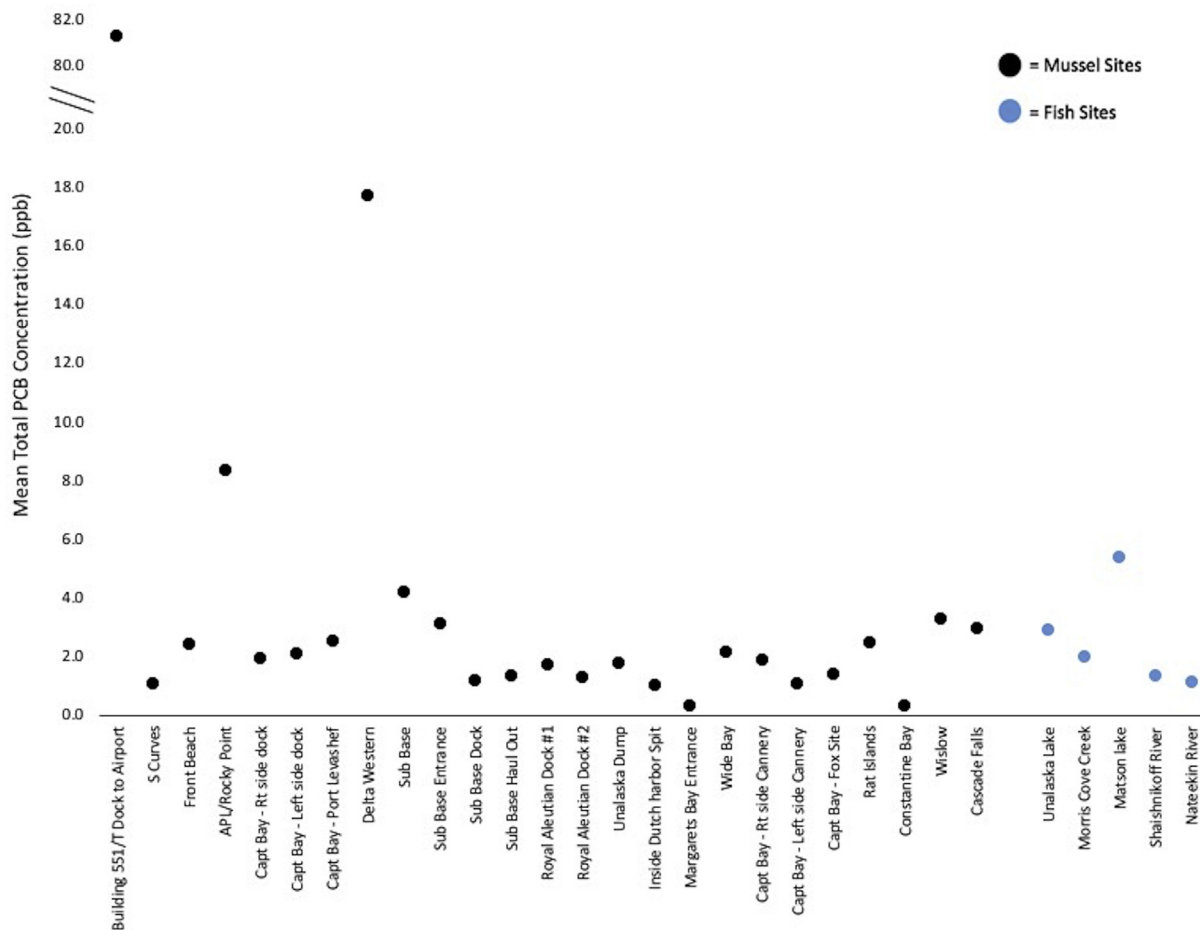


Figure 5. Comparison of mean PCB concentration across all sites. Two FUD sites where mussels were sampled showed levels high above the rest.

3. Results

The mean PCB concentrations across all mussel samples was 6.1 ppb (95% confidence interval, 2.3–11.8 ppb; see Supplemental Data). Mussels from FUD sites had nearly double the PCB concentrations (7.6 ppb) compared to non-military sites (3.9 ppb), a difference of 3.7 ppb, though the 95% confidence interval for this difference overlapped with zero

(-5.3–15.2 ppb; Figure 3). The PCB concentrations found in mussels were especially elevated at two FUD sites: the mean total PCB concentration was 81.4 ppb for mussels collected from “Building 551/T Dock to Airport”, and 17.8 ppb for mussels collected from “Delta Western” (Figures 3 and 5).

The mean total PCB concentration for fish was 2.8 ppb (95% confidence interval, 2.2–3.4 ppb). Fish PCB concentrations were higher at

FUD sites (3.2 ppb) compared to non-military sites (1.2 ppb), a difference of 2.0 ppb (95% confidence interval, 0.12–4.1 ppb; Figure 4).

4. Discussion

Fish collected at FUD sites had higher PCB concentrations than those collected at non-military sites, as did mussels collected from two of the FUD sites (Figures 3, 4, and 5), consistent with a persistent legacy of contaminants from military activities in the region during and after World War II. Due to the extraction efficiency of the QuEChERS method, the resulting PCB levels recovered from Unalaska samples are an underestimation of the total PCB concentrations present. Even with an underestimation, these results demonstrate higher concentrations at military sites and provide site-specific information that can be used for decisions regarding food safety following the U.S. Environmental Protection Agency (EPA) safe consumption criteria for PCBs (EPA, 1999).

The fear of eating potentially contaminated foods and a lack of pertinent data may lead arctic communities to shy away from their traditional subsistence diet. Avoiding the traditional diet in favor of imported Western foods carries health risks given the high nutritional value of the traditional diet, which in coastal communities is primarily marine in origin, vs. the often-lower nutritional value of processed foods (Hardell et al., 2010). More information is needed to determine contaminant concentrations in arctic subsistence foods and variation in contaminant levels associated with local point sources of pollution, such as FUD sites. Two FUD sites of concern for the Qawalangin Tribe are Building 551, a former Dutch Harbor Naval Base Mess Hall, and the Delta Western Fuel Dock across from Building 551 (Figure 2B). During World War II, Japanese forces bombed the port adjacent to Building 551, which damaged transformers and other electrical equipment in the area, leading to PCB contamination of the watershed.

Bootstrapping results for differentiating PCB concentrations of mussels at military vs. non-military sites showed a difference of 3.7 ppb, while the same analysis for fish showed a difference of 2 ppb. PCB concentrations in fish were higher at military sites but PCB concentrations in mussels were only significantly higher in certain military sites where mussels were collected (Figures 3, 4, and 5).

The EPA set risk assessment guidelines for both cancer and non-cancer health endpoints for consuming fish contaminated with PCBs (EPA, 1999). Highly elevated concentrations of PCBs were found in the mussel samples collected at the Building 551 and Delta Western Fuel Dock FUD sites (Figure 5). The PCB concentrations in mussels collected from the Building 551 FUD site fall within the EPA safe consumption guidelines of two meals/month for noncancer health endpoints and 0.5 meals/month for cancer related health endpoints (EPA, 1999). The PCB concentrations in mussels collected from the Delta Western Fuel Dock FUD site fall within the safe consumption limits of eight meals/month for non-cancer health endpoints and two meals/month for cancer related health endpoints. In order to test the food safety across all sites, data were resampled using bootstrapping to determine if the concentrations of PCBs found in mussel and fish samples are above or below consumption guidelines. The overall mean total PCB concentration was 6.1 ppb for mussel samples and 2.8 ppb for fish samples. The concentrations for mussels fall within the 16 meals/month for non-cancer related health endpoints and 4–8 meals/month for cancer related health endpoints (EPA, 1999). The concentrations for fish do not reach a specified meals/month consumption limit for non-cancer related health endpoints and fall within 16 meals/month for cancer related health endpoints (EPA, 1999).

Filter feeding mussels have slightly elevated PCB concentrations compared to surrounding sediments (Bard, 1999). In contrast, due to the lipophilic nature of PCBs coupled with bioaccumulation and biomagnification, high trophic level predators accumulate higher PCB body burdens during their relatively long life spans (Bard, 1999). Therefore, higher trophic level organisms at these sites should have even greater concentrations of PCBs in their tissues. Due to the World War II bombing

at the Building 551 area, elevated PCB concentrations were expected at Building 551 and the Delta Western Fuel Dock FUD sites. These results indicate that these FUD sites should be prioritized for PCB remediation.

In addition to potential adverse health effects for people, PCBs cause detrimental effects in wildlife. For example, bald eagles (*Haliaeetus leucocephalus*) and many other high trophic level birds rely on the near shore marine environment for most of their prey (Elliott et al., 2011). Eagles also forage in landfills exposing them to additional anthropogenic contaminants (Elliott et al., 2006). The Unalaska dump contains a PCB storage area around which eagles and other resident birds forage, potentially exposing them to increased levels of PCBs beyond that obtained in the nearshore marine environment.

5. Conclusions

These results provide preliminary information for land managers, healthcare workers, and the Qawalangin Tribe for the evaluation of level of risk associated with PCB exposure from traditional foods on Unalaska on a site-specific basis. These data may also be employed in prioritizing FUD site remediation on Unalaska. Additionally, the data will assist the U.S. Fish and Wildlife Service in their efforts to manage for contaminant exposure of wildlife within the Alaska Maritime National Wildlife Refuge. Determination of management concerns can be facilitated by the monitoring of important fish prey species of higher trophic level wildlife (Kenney et al., 2012, 2014). PCB data from Unalaska also contribute to the understanding of ecotoxicology in a poorly studied region of the Arctic.

High trophic level organisms that rely on marine food sources, as well as many subsistence communities of the Arctic, can be adversely affected by PCBs and other anthropogenic contaminants (Hardell et al., 2010; Letcher et al., 2010). The presence of PCB contamination in subsistence food species, particularly near certain FUD sites, is a concern for safe consumption on Unalaska. In order to better understand the exposure risks for people and wildlife, further work should investigate the food web dynamics of PCBs and other POPs in both marine and terrestrial habitats on Unalaska.

Declarations

Author contribution statement

Elise M. Adams: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Frank A. von Hippel: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Bruce A. Hungate: Analyzed and interpreted the data; Wrote the paper.

C. Loren Buck: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

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