

**POLLUTANT LOADS AND DISTRIBUTIONS FOLLOWING A MAJOR
FLOODING EVENT IN GALVESTON BAY, TEXAS**

An Undergraduate Research Scholars Thesis

by

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ABSTRACT

Pollutant Loads and Distributions Following a Major Flooding Event in Galveston Bay, Texas

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The inundation associated with flooding events often transfers otherwise abnormal levels of contaminants into the ecosystem. The Houston/Galveston region poses a unique threat due to the high concentration of petrochemical facilities and Superfund sites in the area. High levels of contaminants can disrupt and pollute ecosystem cycling resulting in severe ecological impacts. The resultant flooding of the extreme precipitation from Hurricane Harvey has created a unique opportunity to analyze the release of polycyclic aromatic hydrocarbons, as well as other persistent organic pollutants, from flooded soil in the Houston-area watersheds. Given the petrochemical makeup of the area, trace metals, polycyclic aromatic hydrocarbons, and polychlorinated biphenyls were chosen as the contaminants of concern. Samples were collected from eight locations with varying proximities to four primary Superfund sites and petrochemical plant sites east of Houston, Texas. A Direct Mercury Analyzer was used to determine total mercury concentrations among the sites. Extractions were performed with a Dionex Accelerated Solvent Extractor and the resultant analytes were analyzed for polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) with a Varian GC/MS system. The highest mercury concentration was located downstream of the San Jacinto River

Waste Pits at 25 ppb, which is well below the USEPA guidelines for mercury levels. Total PAH and PCB concentrations ranged from 9.1–371.6 ppb. The predominant PAHs were fluoranthene, pyrene, chrysene, and benzo[a]pyrene. Ratios of fluoranthene/pyrene were consistently greater than 1, indicating a pyrogenic origin of PAHs mostly from coke and coal combustion at a relatively high temperature.

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NOMENCLATURE

COC	Contaminant of concern
DCM	Dichloromethane
DMA	Direct mercury analyzer
EPA	Environmental Protection Agency
GCMS	Gas chromatograph mass spectrometer
Hg	Mercury
Na-tech	Natural hazard triggering technological disasters
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PEC	Probably effect concentration
POP	Persistent organic pollutant
SJRWP	San Jacinto River Waste Pits
SRM	Standard reference material
T-Hg	Total mercury
TCEQ	Texas Commission on Environmental Quality
TEC	Threshold effect concentration

CHAPTER I

INTRODUCTION

Hurricane Harvey generated over 40” of precipitation in the Houston/Galveston region with areas experiencing up to eleven days of inundation. Though Hurricane Harvey has yet to be fully quantified, it is projected to have caused not only economical, but ecological damages comparable to those of Hurricane Katrina. Various ecological hazards from Katrina include: increased and potentially detrimental levels of mercury, arsenic, lead, and chromium, over 6.5 million kg of hazardous anhydrous ammonia released in the air, and various fuel leakages from petrochemical facilities (Harmon and Wyatt, 2008; Picou, 2009; Presley et al., 2006). The low lying locations of Houston’s Superfund sites coupled with the presence of persistent organic pollutants (POPs) create a catastrophic potential with the capacity to be severely exacerbated by extreme climatic events.

Harris and Galveston counties alone house 29 Superfund sites and 30-50% of the nation’s petrochemical and industrial facilities (TCEQ, 2018; EPA, 1980). Additionally, the area is largely a densely populated coastal flood plain, making the region a model for natural hazard triggering technological disaster (na-tech) events (FEMA, 2017). With increasing frequency and severity of natural hazards such as hurricanes, the redistribution and release of legacy contaminants through sediment disruption in the Houston-Galveston region poses a unique threat. The most recent of these natural disasters in the area, Hurricane Harvey, produced the largest rainfall documented in the U.S. generating record breaking flooding along Buffalo Bayou and the San Jacinto River (NOAA, 2017)(Figures 1 and 2).



Figure 1. San Jacinto River and Buffalo Bayou before Hurricane Harvey (Copernicus, 2017).



Figure 2. The resultant flooding from Hurricane Harvey along San Jacinto River and Buffalo Bayou as of August 30th, 2017 (Copernicus, 2017).

The remobilization of legacy contaminants through sediment disruption extends their presence in the environment and therefore increases the concentration of POPs active in ecosystem cycling (TCEQ, 2017). The Superfund sites proximal to the locations sampled historically contain large concentrations of various POPs (EPA, 2017) (Figure 3).

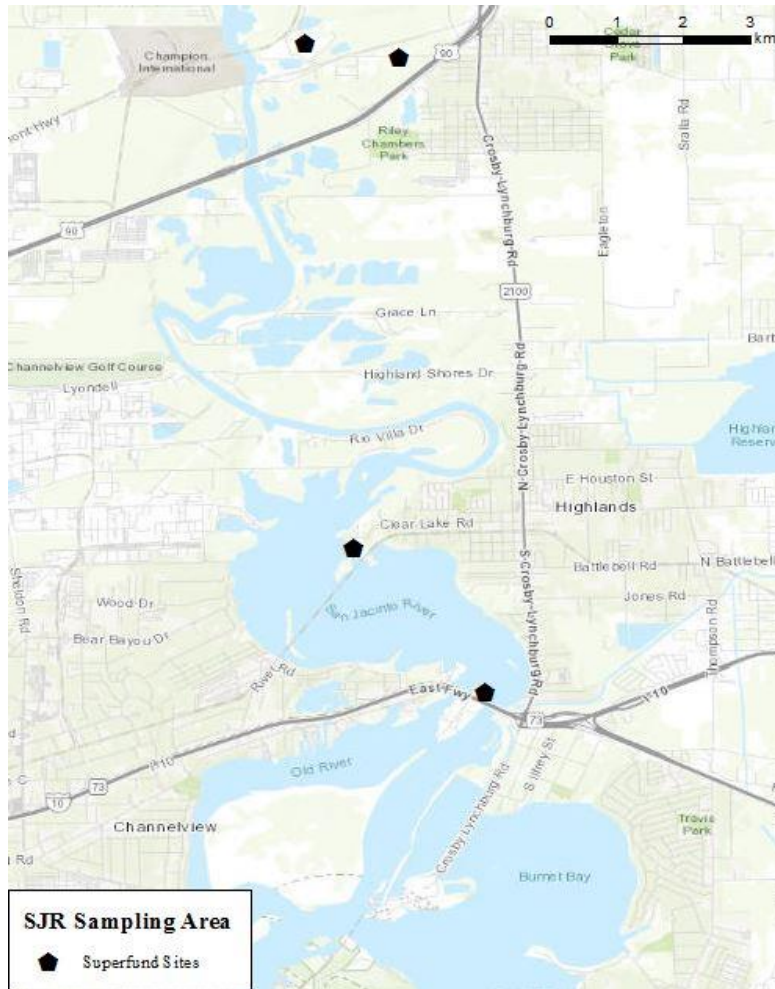


Figure 3. Map east of Houston with the relevant Superfund site locations in black.

Located in a residential area, French Limited is a 55-acre site that borders neighborhoods and a public marina near the San Jacinto River. Due to a lack of regulation, exact quantities of contaminant loads for the Superfund sites are unknown. However, it is estimated as much as 70

million gallons of industrial waste from petrochemical facilities containing heavy metals, polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyls (PCBs) were dumped at the French Limited site from 1966 to 1971 (EPA, 2018). The site was active between 1950 and 1973, making the total gallonage likely even higher. With the marina and San Jacinto River as the west border, just north of French Limited and south of Jackson Bayou lies the 185 acre Sikes Superfund site. Sikes was an illegal “open dump” from 1961 to 1967, meaning it allowed bulk waters to be discarded directly into the soil. Approximately 2,000 55-gallon drums were dumped on the site along with an indeterminable amount of bulk loads containing heavy metals, PCBs, chloroform, and various other contaminants (EPA, 2017). Located at the end of a residential peninsula in the San Jacinto River, the Highlands Acid Pit was used as a discharge area for oil refineries with large concentrations of heavy metals found in 1981, which have since then been mitigated (TCEQ, 2018). The San Jacinto River Waste Pits (SJRWP), located in and along the river itself, have been cited as dioxin point sources. In a recent study, models and dye tracing were used to identify SJRWP as the origin of much of the dioxin concentration found in the Galveston Bay system (Rifai, 2016). In addition to dioxins, SJRWP has been linked to PCB, PAH, and heavy metal concentrations in riverine and estuarine sediments, leading to seafood advisories up and downstream of the site. A recent remediation review confirmed the dispersal of these contaminants through flooding events (Garland, 2015).

With the abundance of Superfund sites and petrochemical facilities present in the Houston/Galveston area, the primary focus for the analyses will be on POPs including heavy metals, PCBs, and PAHs. POPs are persistent in the environment and bio-accumulate, making even low concentrations detrimental to ecosystem health. Mercury concentrations set a baseline for general estuary health and serves as an indicator of the presence of more toxic mercury

species, such as methyl mercury, and will therefore be the contaminant of concern (COC) for heavy metal analysis. For Texas, the general background concentration of mercury is approximately 40 parts per billion (ppb) and is not considered harmful until the concentration approaches 486 ppb. PAHs and PCBs are hydrophobic, carcinogenic, and mutagenic, making them a particularly nefarious environmental concern (Ingersol et al., 2000). Furthermore, these compounds bioaccumulate and concentrations as low as 0.5 ppb are above the USEPA guidance levels for residential areas. The Houston Ship Channel has historically housed the highest concentrations of PCBs, even as related to areas of comparable industrial quality, and thus the region contains major pollutant potential (Howell et al., 2008).

With the magnitude of precipitation and long-term inundation due to Harvey coupled with the industrial make-up of the Houston/Galveston region, there is a potential increased risk of the remobilization and release of legacy contaminants into the ecosystem. The determination of concentrations and distributions of POP loads released during Harvey will provide a detailed analysis of impact from floodwaters and create a high-resolution data set of the contaminants ultimate fates.

CHAPTER II

SAMPLING AND METHODS

Data collection

Sampling locations were based on proximity to Superfund sites, major flooding areas, and legal accessibility. Two field runs were performed east of Houston along major waterways for soil sample collection in October 2017. A total of 28 bagged soil samples from various locations were collected (Figure 4) and stored at 4°C until processing.

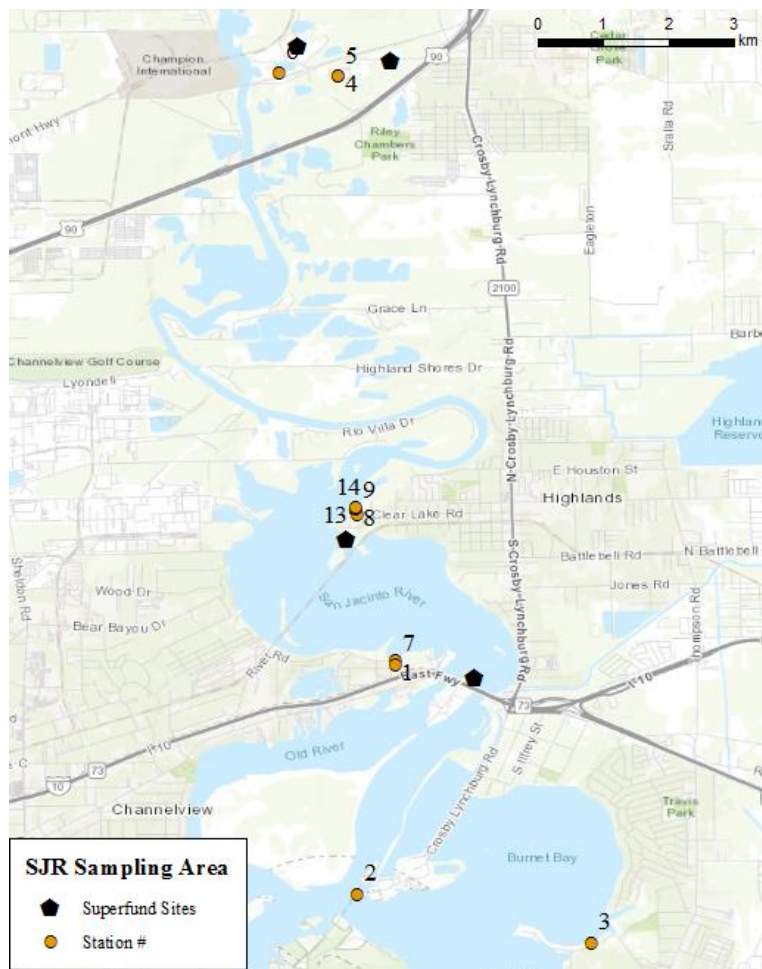


Figure 4. Map of sample locations with their respective station numbers.

Mercury concentrations

Prior to total mercury (T-Hg) analysis, the samples were dried at 50°C for five days, ground, and homogenized. A Milestone DMA 80 automatic Hg analyzer was used in accordance to EPA Method 7473 (EPA, 1998). A standard reference material (SRM), marine sediment SRM MESS-3, with a known T-Hg concentration was used to create a five-point calibration curve. A 0-100 mg range was used and verified by the SRM results for the certified range. Approximately 20 mg of each sample were loaded in sample boats and introduced to the analyzer, drying for 300 seconds at 200°C with thermal decomposition for 180 seconds at 650°C.

PAH and PCB concentrations

Extractions were performed using a Dionex Accelerated Solvent Extractor (ASE 200). Approximately 15 g of each ground sample were loaded into 22 mL extraction cells with 5 g of purified sand. The cells were placed in the autosampler tray and a 1:1 dichloromethane(DCM):acetone solvent was used to perform the extractions. The operational parameters were as follows: a 5 min preheat to 100°C with a 5 min heat time at 1500 psi, followed by a 5 min static period. Pressurized argon gas was used to purge for 60 seconds and the resulting analytes were evaporated to dryness.

The dried samples were redissolved in 1:1 DCM:acetone solvent and analyzed with a Varian 4000 GCMS system with an ion mass detector. The internal standards used for PAHs and PCBs were d12-perylene and PCB 65, respectively. In splitless mode, separation was performed with an Agilent DB-5 capillary column and helium as the carrier gas with a 1ml min⁻¹ flow rate. The temperature program was set to delay for 3 min, increase to 40°C and hold for 1 min, increase to 180°C at a rate of 20°C min⁻¹, and increase to 300°C at 5°C min⁻¹ and hold for 10 min.

Using the retention times, quantitative ions, and qualitative ions in, the samples were analyzed for the concentrations of the respective PAHs and PCBs (Table 1).

Table 1. Retention times, quantitative ions, and qualitative ions used to determine the presence and concentrations of PAHs and PCBs.

Compound	Retention time (mi)	Quant. Ion	Qual. Ion
Napthalene	6.68	128	102
Acenaphthylene	8.66	152	76
Acenaphthene	8.92	153	76
PCB 1	8.97	188	152
Fluorene	9.78	166	139
PCB 18	11.78	186	256
Phenanthrene	11.91	178	152
Anthracene	12.05	178	152
PCB 65*	14.04	297	225
Fluoranthene	15.68	202	101
PCB 101	16.36	326	254
Pyrene	16.47	202	101
PCB 138	19.88	360	290
Benzo[a]anthracene	21.46	228	114
Chrysene	21.59	228	113
PCB 180	22.08	394	324
Benzo[b]fluoranthene	25.91	252	126
Benzo[k]fluoranthene	26.04	252	126
d12-Perylene*	27.04	264	260
Benzo[a]pyrene	27.12	252	126
Indeno[1,2,3-cd]pyrene	31.15	276	207
Diben[a,h]anthracene	31.3	278	207
Benzo[g,h,i]perylene	31.96	276	207

*Internal standards used for GCMS analysis.

CHAPTER III

RESULTS

T-Hg analysis

Overall low concentrations of T-Hg were found at every location ranging from 2.248-25.305 $\mu\text{g}/\text{kg}$ (Table 2). The majority of locations contained samples below 5 $\mu\text{g}/\text{kg}$, with a few exceptions that were inconsistent with any latitudinal or proximity patterns.

Table 2. T-Hg concentrations across all locations.

Station	Latitude	Longitude	T-Hg [$\mu\text{g}/\text{kg}$]
#1	29.797224	-95.073479	16.724
#2	29.764952	-95.078576	11.201
#3	29.758264	-95.046417	8.559
#4	29.877555	-95.081334	2.692
#5	29.877555	-95.081334	15.951
#6	29.878060	-95.089310	3.483
#7	29.796545	-95.073306	22.971
#8	29.817785	-95.078800	2.354
#9	29.817785	-95.078800	2.302
#10	29.817785	-95.078800	2.478
#11	29.817785	-95.078800	2.722
#12	29.817130	-95.078566	25.305
#13	29.818042	-95.078824	4.885
#14	29.818193	-95.078868	2.248

The T-Hg distribution was heterogeneous across the sampled regions and no trends were apparent. Though samples were taken with varying distance in respect to the Superfund sites, no

correlation was observed (Figure 5). This lack of correlation is especially apparent in samples 8-14, with T-Hg levels varying the full range of concentrations found within almost equal proximity to the Highlands Acid Pit.

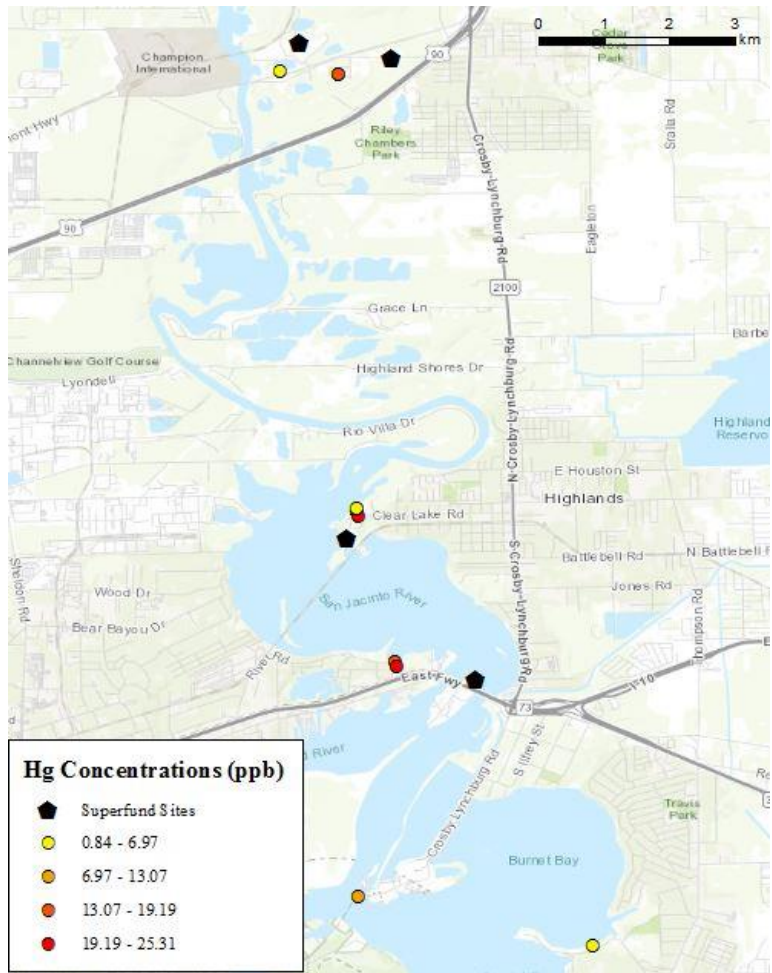


Figure 5. Sampling sites with gradient mercury concentrations.

PAH and PCB analysis

PAHs fluoranthene, pyrene, chrysene, and benzo[a]pyrene were found at nearly every sampled location. Outside of these primary PAHs, the concentration for each station was

irregular. The most consistent distribution was found between stations 1, 4, 6, and 7 which only contained the primary PAHs (Figure 6).

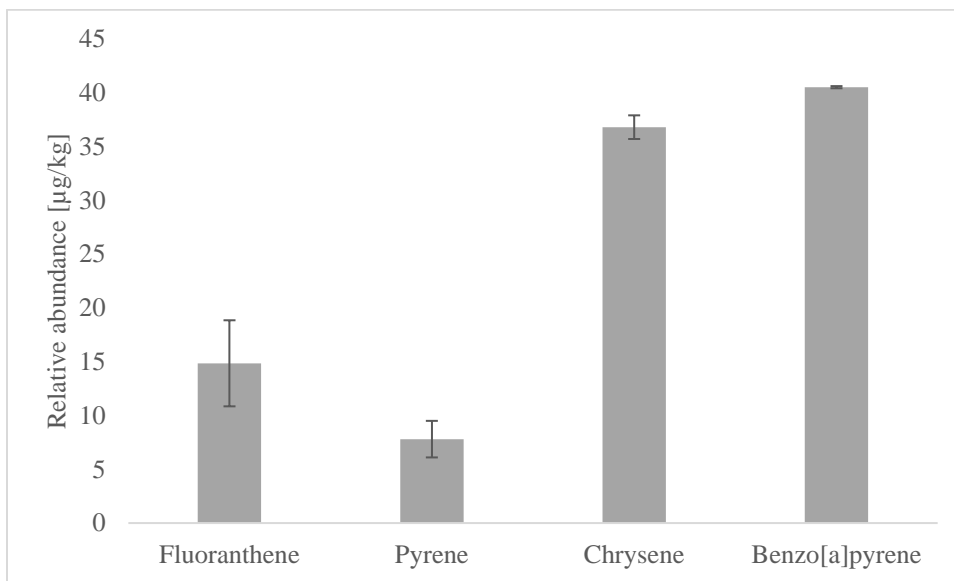


Figure 6. Relative abundance of PAHs across stations 1, 4, 6, and 7.

The ferry station contained six PAHs that were not present at any other location. Though found at every tested location, overall PAH concentrations were consistently low (Table 4). With only two exceptions, sum concentrations at each site were below 100 µg/kg. The location with the highest total PAH concentration was station 2, the shore by the Lynchburg Ferry landing (Table 4). No clear relationship was apparent between PAH concentration and proximity to a Superfund site source, nor between the concentration and the source being up or downstream from the station. No latitudinal correlation was evident as demonstrated by stations 2 and 3 exhibiting both extremes of the concentration range (Figure 7).

Table 4. PAH concentrations in [$\mu\text{g}/\text{kg}$], or ppb.

Station	Napthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benzo[a]anthracene	Chrysene	Benzo[b]fluoranthene	Benzo[k]fluoranthene	Benzo[a]pyrene	Indeno[1,2,3-cd]pyrene	Diben[a,h]anthracene	Benzo[g,h,i]perylene	ΣPAH
#1	bd	bd	bd	bd	bd	bd	19.5	9.1	bd	33.7	bd	bd	35.0	bd	bd	bd	97.3
#2	bd	25.6	bd	1.3	bd	bd	91.4	64.0	bd	bd	40.5	5.2	43.1	14.4	60.1	26.1	371.6
#3	bd	bd	bd	bd	bd	bd	12.9	6.5	bd	bd	bd	bd	bd	bd	bd	bd	19.4
#4	bd	bd	bd	bd	bd	bd	11.7	6.5	bd	31.3	bd	bd	35.2	bd	bd	bd	84.7
#6	bd	bd	bd	bd	bd	bd	10.4	5.2	bd	31.3	bd	bd	35.2	bd	bd	bd	82.1
#7	bd	bd	bd	bd	bd	bd	12.9	7.8	bd	32.3	bd	bd	34.9	bd	bd	bd	88.0
#8	bd	bd	bd	bd	bd	bd	9.1	bd	bd	bd	bd	bd	bd	bd	bd	bd	9.1
#9	bd	bd	bd	bd	bd	bd	9.1	bd	bd	bd	bd	bd	35.3	bd	bd	bd	44.4
#11	bd	bd	bd	bd	bd	bd	9.1	5.2	bd	bd	bd	bd	35.2	bd	bd	bd	49.5
#12	bd	bd	bd	bd	bd	bd	18.3	9.1	bd	33.9	bd	bd	52.2	bd	49.6	bd	163.2
#13	bd	bd	bd	bd	bd	bd	10.5	5.2	bd	31.4	bd	bd	35.3	bd	bd	bd	82.3
#14	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd	bd
																	1091.6

*bd: below detection

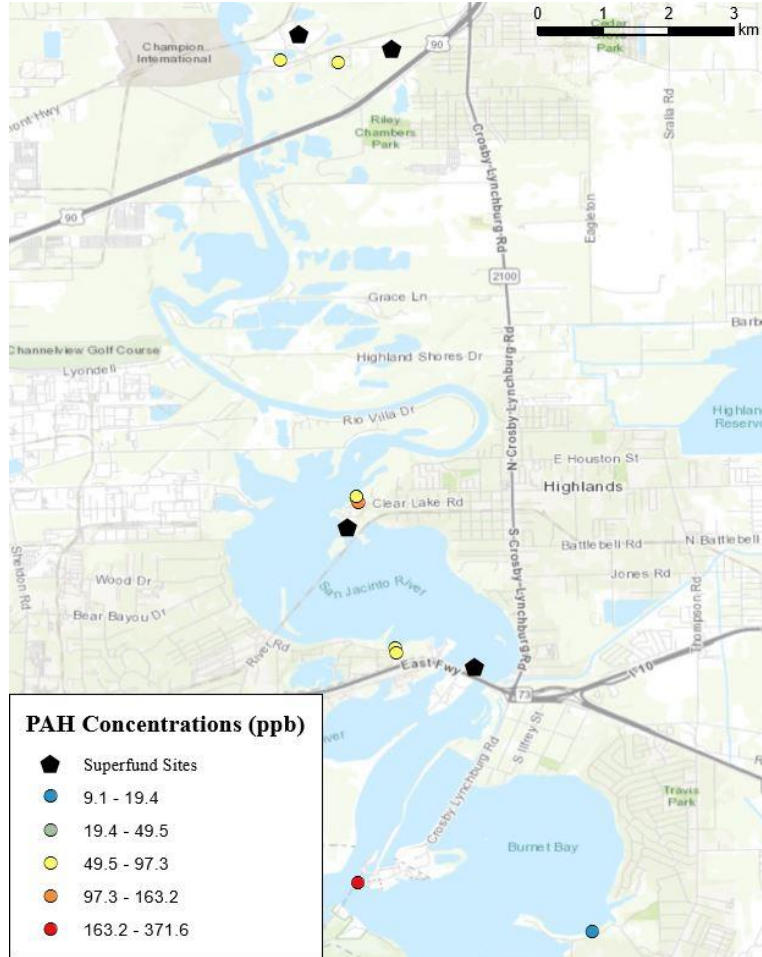


Figure 7. Map of sampled region with gradient PAH concentrations in [$\mu\text{g}/\text{kg}$], or ppb.

Only one PCB concentration was detected at one station (Table 5). The concentration was relatively low at $24.6 \mu\text{g}/\text{kg}$. Subsequently, no latitudinal or proximity trends were ascertained (Figure 8).

Table 5. PCB concentration from the sampled region.

Station	PCB 1 [$\mu\text{g}/\text{kg}$]	PCB 18 [$\mu\text{g}/\text{kg}$]	PCB 101 [$\mu\text{g}/\text{kg}$]	PCB 138 [$\mu\text{g}/\text{kg}$]	PCB 180 [$\mu\text{g}/\text{kg}$]	ΣPCB [$\mu\text{g}/\text{kg}$]
#3	bd	bd	24.6	bd	bd	24.6

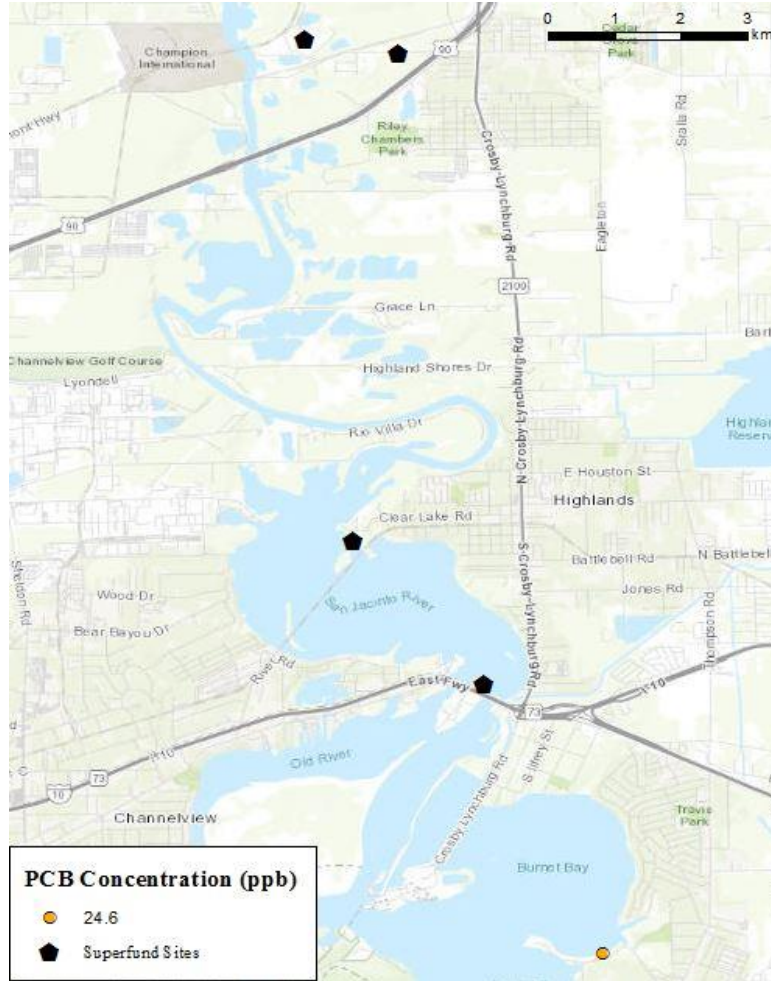


Figure 8. Map of sampled region with PCB concentration.

T-Hg correlation with PAHs

A weak correlation was observed between T-Hg and PAH concentrations. With increased T-Hg concentration a moderate trend was evident of PAH concentration also increasing (Figure 9).

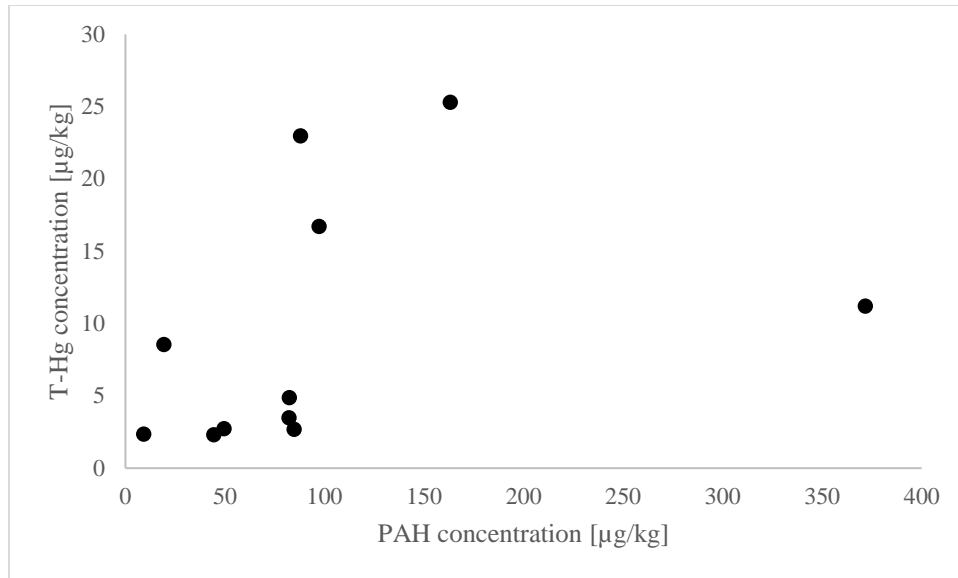


Figure 9. Correlation between T-Hg and PAH concentration.

PAH ratios

One significant PAH ratio was found. Concentrations of fluoranthene were consistently higher than pyrene concentrations and the ratio of fluoranthene to pyrene was therefore consistently greater than one (Table 6).

Table 6. Ratio of fluoranthene to pyrene concentrations.

Station	Fluoranthene µg/kg	Pyrene µg/kg	F:P ratio
1	19.5	9.1	2.1
2	91.4	64	1.4
3	12.9	6.5	2.0
4	11.7	6.5	1.8
6	10.4	5.2	2.0
7	12.9	7.8	1.7
8	9.1	bd	NA
9	9.1	bd	NA
11	9.1	5.2	1.8
12	18.3	9.1	2.0
13	10.5	5.2	2.0
14	bd	bd	NA

CHAPTER IV

DISCUSSION

Hurricane Harvey devastated many regions in the Gulf Coast. The large number of refineries, chemical plants, and flood-prone legacy waste sites within the Houston watershed shaped a distinctive vulnerability to natural events in the Houston-Galveston region. The San Jacinto River and surrounding water bodies make up a tidally dominated system in a heavily populated area abundant in industrial facilities, granting the region catastrophic economic and ecological potential. A comparable event, Hurricane Katrina, produced PAH-saturated floodwaters that inundated Louisiana, and resulted in a redistribution of sediment-associated legacy contaminants from Superfund sites (Picou, 2009). Similarly, Hurricane Ike caused an influx in PCB concentrations seen in sediment samples (Howell and Rifai, 2015). Therefore, when Hurricane Harvey's floodwaters inundated areas around Superfund sites, it was expected to see a redistribution of legacy contaminants into the ecosystem, as evidenced through various cases. Though reinforced in 2014, damage to the armored cap on the SJRWP was confirmed by the EPA after Hurricane Harvey (Brody et al., 2014; EPA, 2017). The resultant exposure of underlying contaminants to the surrounding area produced dioxin levels up to 70 $\mu\text{g}/\text{kg}$, several magnitudes above the EPA recommended concentration of 0.03 $\mu\text{g}/\text{kg}$ (EPA, 2017). Hurricane Harvey also appeared to have redistributed liquid mercury onto a property near the SJRWP Superfund site, suggesting the presence of heavy metals in dangerous concentrations and the potential for significant toxicity (Healy and Kaplan, 2017).

Concentrations and distributions of T-Hg

Mercuric chloride (HgCl_2) is an inorganic form of mercury that is pervasive in ecosystem cycling and frequently acts as a source of methylation, resulting in alkyl species (Bloom et al., 1999). Alkyl mercury species are highly mobile through environmental processes, which inevitably leads to bioaccumulation of the more toxic speciation (Han et al., 2003). T-Hg levels serve as a baseline for the possible presence of toxic mercury species and act as useful indicators for ecosystem health. The threshold effect concentration (TEC) is the level to which a contaminant can be present without any expected significant effect on an ecosystem. T-Hg concentrations were low overall (Table 2) and all below the TEC of $180 \mu\text{g}/\text{kg}$ (MacDonald et al., 2000). The highest level of T-Hg was a mere $25.305 \mu\text{g}/\text{kg}$ with even lower concentrations around $5 \mu\text{g}/\text{kg}$ as the norm (Table 2). These concentrations were consistent with median background levels and were even below what recent studies have found in waters from Galveston Bay at Trinity River (Connor et al., 1975; Matsumoto et al., 2010). The distribution of T-Hg was heterogeneous, and no latitudinal or potential source proximity correlations were observed (Figure 5). An indiscriminate dispersal has been shown to be consistent with natural distributions, indicating the concentrations found were representative of background levels of T-Hg.

Concentrations and distributions of PAHs and PCBS

PAHs and PCBs are hydrophobic contaminants with an affinity for binding to organic matter. This characteristic creates pollutants that are persistent in the environment and leads to the bioaccumulation of carcinogenic, mutagenic toxins. Relatively minimal concentrations of PAHs and PCBs are considered environmentally detrimental due to their adverse properties. The

concentrations of individual PAHs were consistently low with levels below the TEC and well below the probable effect concentration (PEC)(Table 4). The sum of concentrations was 1091.6 $\mu\text{g}/\text{kg}$. Though still relatively low, no guideline has been set for combined PAH concentration, likely due to the limited knowledge of interactions between the various PAHs and environment (MacDonald et al., 2000). The four most common PAHs found were congruous with PAHs commonly affiliated with combustion: fluoranthene, pyrene, chrysene, and benzo[a]pyrene (Yunker et al., 2002). The most consistent distribution was seen between sampling locations 1, 4, 6, and 7 likely because only the most common PAHs were present at these sites (Figure 6). Location 2 had six PAHs that were not present at any other sampling site (Table 4), however, these PAHs were still consistent with a combustion affiliation (MacDonald et al., 2000). The proximity of location 2 to a ferry landing and the subsequent presence of an increased source of combustion due to idling vehicles indicates the source of PAHs was unlikely from legacy contaminants. With no observed proximity or latitudinal patterns with respect to the Superfund sites, no source or legacy redistribution conclusions were made. However, PAH ratios are valuable tools in source characterization (Stogiannidis and Laane, 2015). The ratio of fluoranthene to pyrene was consistently greater than one, indicating coke and/or coal combustion as the likely source (Stogiannidis and Laane, 2015)(Table 6). Only a concentration of PCB 101 (24.6 $\mu\text{g}/\text{kg}$) was found and no patterns could be ascertained (Table 5). The concentration of the PCB was below the TEC of 59.8 $\mu\text{g}/\text{kg}$.

Considering the high number of petrochemical facilities in the Houston-Galveston region, the persistently low concentrations were not expected. Historically, Houston waters have contained an unusually high level of PCBs and would therefore be expected to distribute them during flooding events (Howell et al., 2008; Garland, 2015). With similar natural disasters

leading to the release and remobilization of legacy contaminants, a more complete analysis is needed as suggested by this survey's inconsistencies with previous findings. A weak correlation was observed between T-Hg and PAH concentration (Figure 9). This could be indicative of various relationships such as disposal history, hydrological patterns, or shared physical characteristics between the sampling locations and is a useful direction for future experimentation. With the abundance of potential contaminant sources in the area, low POP concentrations were unexpected, but could possibly be due to the floodwaters themselves. Flood mobilization could have led to an influx of organic carbon, fueling bacterial metabolism and resulting in efficient biodegradation of PAHs. The low concentrations could very well imply successful mitigation techniques, however, it is more likely the contaminant levels were the result of sampling from fresh soil locations rather than marine. The projected increase in severity and frequency of natural disasters necessitates a deeper understanding of contaminant remobilization for this na-tech disaster prone region. The unexpected results highlight the demand for a more thorough analysis and expose compelling potential pathways to investigate.

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