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PFAS Contamination in WNC Waterways

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Abstract

Per and polyfluoroalkyl substances (PFAS), a group of over 4,700 man-made compounds that have been produced beginning in the 1950s, have become widespread environmental contaminants. PFAS compounds have been found ubiquitously in the environment and have been found in the blood of 97% of Americans.Past research done by the Brock Lab group (Henry Hill and Steven Defigila) adapted a solid phase extraction method (EPA 533) to extract PFAS compounds from water. The work by Henry Hill and Steven Delfugia found that surface waters near areas of routine fire fighting practice often had higher concentrations of PFAS. Recently, concerns have arisen about the presence of PFAS compounds in artificial turf fields. The presence of PFAS compounds in the fields could potentially be damaging to the health of people who use or live downstream of these fields. The current project sampled bodies of water located downstream of artificial turf fields in order to see if a correlation exists between artificial turf and surface water PFAS contamination. Several locations were found with combined PFAS levels above 10 ppt. Six PFAS compounds (8:2FTS, 4:2FTS, PFHxS, PFBA and PFOS) were found in more than one location with artificial turf. Extractions were also done on samples of artificial turf from different brands. The first round of extractions, done in pure water with glacial acetic acid, did not show PFAS contamination. The second round of extractions done in 50% water and 50% ACN have not been analyzed at this time.

Introduction

Per and Polyfluoroalkyl substances (PFAS), a class of purely synthetic compounds, have been manufactured since the 1950s.[1] PFAS compounds are often referred to as forever chemicals due to their inability to break down in the environment. When degradation occurs, shorter chain PFAS compounds are created from the longer ones. PFAS can be broken into two main categories: perfluorinated carboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs); consisting of over 4700 unique compounds that are thermally stable, lipophilic, and do not break down causing bioaccumulation within in the environment.[2] PFAS are persistent in the environment due to the strength of the carbon-fluorine bond; microbes are unable to digest PFAS compounds. PFAS are used in many different products such as firefighting foams, non-stick pans, plastics, waterproof materials, stain resistant carpets, textiles[1], and makeup[3].

The most widespread PFAS compounds are PFOS (Perfluorooctane Sulfonate) and PFOA (Perfluorooctanoic acid) commonly referred to as legacy compounds. Legacy compounds were the first PFAS compounds in production. PFOS was phased out of production in 2002 due to health concerns regarding the compound; PFOA was phased out in 2015 for the same reasons. After the phase out, new types of PFAS were created to replace the legacy compounds. These compounds are referred to as“next generation” PFAS compounds.[1]

Table 1. Selection of PFSA compounds

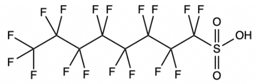
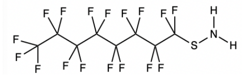
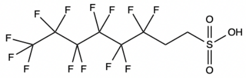
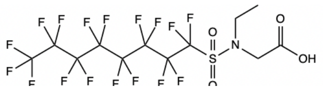
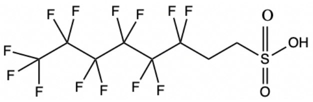
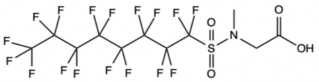
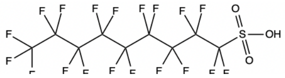
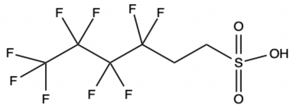
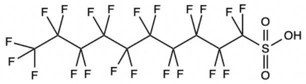
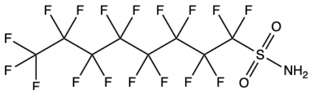
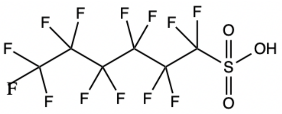
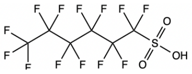
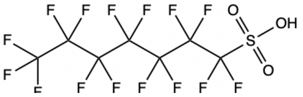
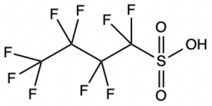
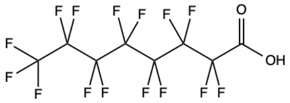
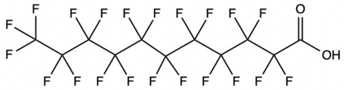
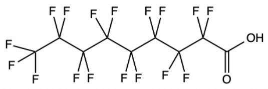
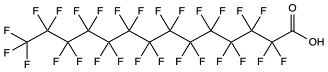
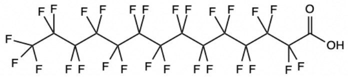
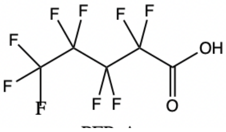
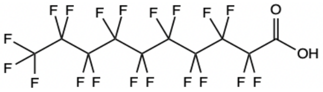
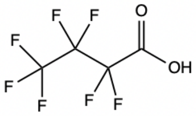
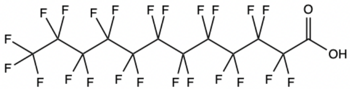
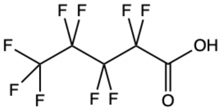
Name	**Structure**	**Name**	**Structure**
Perfluorooctane Sulfonate (PFOS)		Perfluorooctane sulfonamide (FOSA)	
Fluorotelomer sulfonic acid 8:2 (8:2 FTS)		2-(N-Ethyl perfluorooctane sulfonamido) acetic acid (N-Et-FOSAA)	
Fluorotelomer sulfonic acid 6:2 (6:2 FTS)		2-(N-Methyl perfluorooctane sulfonamido) acetic acid (N-Me-FOSAA)	
Perfluorononane sulfonic acid (PFNS)		Fluorotelomer sulfonic acid 4:2 (4:2 FTS)	
Perfluorodecane sulfonic acid (PFDS)		Perfluorooctane sulfonamide (PFOSA)	
Perfluorohexane sulfonic acid (PFHxS)		Perfluorohexane sulfonic acid (PFHxS)	
Perfluoroheptane sulfonic acid (PFHpS)		Perfluorobutane sulfonic acid (PFBS)	

Table 2. Selection of PFCA Compounds

Name	**Structure**	**Name**	**Structure**
Perfluorooctanoic acid (PFOA)		Perfluoroundecanoic acid (PFUnA)	
Perfluorononanoic acid (PFNA)		Perfluorotetradecanoic acid (PFTeA)	
Perfluoroheptanoic acid (PFHpA)		Perfluoropentanoic acid (PFTrA)	
Perfluoropentanoic acid (PFPeA)		Perfluorodecanoic acid (PFDA)	
Perfluorobutanoic acid (PFBA)		Perfluorododecanoic acid (PFDoA)	
Perfluoropentanoic acid (PFPeA)			

The adverse human health effects of PFAS as a class group are largely unknown. High serum PFNA concentrations (0.70-3.50 ug/L) in humans exposed through drinking water correlated with a 6.41% increase of T4 (thyroxine) production in women. This can increase the likelihood for hyperthyroidism to develop.[4] Concentrations of (35.7-77.3 ug/L) PFOS, (0.70-0.3.50 ug/L) PFNA, (4.10-34.3 ug/L) PFHxS, and (0.20-0.90 ug/L) PFDeA were associated with decreased kidney function.[5] PFOA levels in human semen (0.67 ng/mL) correlated with lower sperm count.[6] There is also a positive association (ratio of 1.62 and 1.54 respectively) between high concentration of PFOS (32.9, 34.1, 30.8 ng/mL) and PFOA (4.37, 4.94, 4.22 ng/mL) in human serum and type 2 diabetes.[24] A study conducted on a human population exposed to high levels of PFOA PFAS (PFOA (65.6 ng/mL) through drinking water experienced an increased death ratio (1.60).[7] This means that the chances of death were elevated by 60%.

Routes of human exposure PFAS include ingestion of contaminated food (specifically seafood[8] and popcorn[9]), contaminated water, using products that contain PFAS, and inhalation of contaminated air[1]. Ingestion exposure is primarily due to the consumption of produce grown in contaminated soil or watered with contaminated water.[10] Surface and groundwater contamination, which affects drinking water, is a result of runoff of products containing PFAS like AFFFs, landfill leachate, as well as manufacturing waste like textiles or plastics. AFFF usage is found around airports, military bases, and fire fighting training facilities.[2]

News sources in the past two years have raised concerns about the presence of PFAS in artificial turf fields.[19] [20, 21] The presence of PFAS in artificial turf fields has raised concern due to the aforementioned health effects of PFAS compounds to the people, often children, who use these fields for recreation. There is also a patent out that requires PFAS in artificial turf[22]. The exact reason why PFAS is used in artificial turf is unknown at this time. A study done in Stockholm, Sweden found that PFAS were present in the backing of artificial turf at low levels. Nine PFAS compounds were targeted, consisting of PFHxA ,PFHpA ,PFOA, PFNA ,PFDA ,PFHxA

,PFDoDA ,PFHxA and PFOS[26]. This project will focus on the possibility of PFAS collecting in water underneath or around artificial turf fields. Water samples near artificial turf fields will be collected and analyzed for PFAS compounds using high performance liquid chromatography tandem triple quadrupole mass spectrometry (LC/MS-MS). In order to determine which PFAS compounds are used in artificial turf, a series of experiments testing different types and brands of artificial turf will be performed.

Background Literature

PFAS compounds are incredibly widespread in the environment. PFAS have been found in the blood of 97% of Americans (NHANES). It is estimated that 18-80 million people in the United States drink water with a combined PFOA and PFOS concentration of 10 ng/L or higher.[11]Drinking water contamination is considered to be a major predictor of PFAS serum levels.[10,13] PFAS compounds have also been found in US bottled water, with higher concentrations in bottles labeled "spring".[14] PFOS (30.4 pg/mL) and PFOA (13.9 pgm/L) had the highest concentrations of PFAS compounds detected in breast milk. PFHxA and PFHpA were also detected in the majority of breast milk samples with concentrations of (9.69 pg/mL) and (6.10 pg/mL).[15] Varying levels of PFAS contamination have been found in surface water across the United States. The PFAS concentration of the entrance of Lake Mead in Las Vegas, Nevada PFAS was measured to be 271.9 ng/L.[16] In New Jersey, concentrations of PFAS in surface water ranged from 22.9 ng/L to 279.5 ng/L.[17] PFAS concentrations ranging from 1.1 ng/L to 74.9 ng/L were measured in surface waters in Washington state.[18] PFAS surface water contamination is not limited to the United States, similar studies have found detectable levels of PFAS contamination in many places around the world.

At this time, very little research exists on PFAS in artificial turf. A study was done in Stockholm Sweden found PFAS at all tested sites with artificial turf. They also concluded that the PFAS present in artificial turf is used in the backing of the turf as opposed to the blades themselves. PFCA compounds were detected more frequently than PFSA compounds.

The Cape Fear River, which is the drinking water supply for 250,000 people, was found to be polluted with GenX in 2017. Gen X is a next-generation PFAS compound. The pollution of the Cape Fear River with Gen X sparked an investigation with the goal of finding out what other compounds had been disposed of in the river. These investigations found a plethora of PFAS compounds throughout the Cape Fear River basin. Research expanded to encompass more of eastern North Carolina. Despite the investigation of PFAS in surface water in eastern of North Carolina, little research exists on quantifying PFAS in western North Carolina.[23]

Previous research by the Brock lab group (Steven Defiglia and Henry Hill) adapted a solid phase extraction method (EPA 533) to extract PFOS from water, as well as a method for analysis and quantification using LC/MC-MS. In this method, a C18 (reversed phase column) was used with a gradient of 20 mM ammonium acetate and methanol. After separation through the column, the sample is ionized via Taylor cone electrospray ionization. Then the sample is propelled through the instrument. The first section of a triple quadrupole mass spectrometer is a mass filter composed of four poles. These poles alternate in positive and negative charges, creating a three dimensional maze. This maze filters ions by mass; ions that are heavier or lighter than the selected precursor ion, crash into the poles and are obliterated. The second section is a collision chamber in which the ions collide with Ar or N_2 gas. This creates fragments called product ions, which then pass into the third chamber. The third chamber is another mass filter that only allows certain ions to reach the detector. LC/MS-MS is highly selective for the analyte in interest, and can detect at parts per trillion levels.

Methodology

In order to reduce potential contamination, lab equipment that could come in contact with PFAS was made out of polypropylene (PPE) or high-density polyethylene (HDPE).

Site Selection and Sampling

Sites were selected based on two critea: presence of articifal turf fields and proximity to surface water. Several sites located near surface water containing sports fields without artificial turf were selected as controls. Before each sampling trip, a field blank consisting of deionized water stored in a 1 L HDPE bottle was prepared. Samples were collected in 1 L HDPE bottles and then stored on ice until returned to the lab. Once in the lab, samples were denatured with 5 mL of 35% nitric acid. Samples were refrigerated until time of extraction.

Solid Phase Extraction (SPE)

All labware used for multiple extractions was rinsed with methanol between each extraction.

A 250 mL aliquot of the water sample was measured using a PPE graduated cylinder. Then 2.5 mL of glacial acetic acid was added to the sample to adjust the pH. Next 200 µL of PFAS internal spiking solution was added to the sample. Afterwards, the sample was filtered through a glass fiber membrane using a miniature Buchner funnel into a polypropylene vacuum flask. To precondition the SPE cartridge, 5 mL of 5% ammonium hydroxide in 60:40 acetonitrile/methanol solution, 5 mL of deionized water and 6 mL of 1% acetic acid in deionized water were passed through an Oasis weak ion exchange (WAX) cartridge. The preconditioned cartridge and barrel were moved to a vacuum flask and the previously prepared sample was passed through the cartage at 10 mL/min. The PPE vacuum flask containing the sample was rinsed twice with 7.5 mL of deionized water to rinse off PFAS at the bottom of the container. The cartridge and barrel were then moved to the manifold and dried at 10-15 mm Hg for 5 minutes. PFAS were eluted from the carriage by adding 8 mL of 5% ammonium hydroxide in 60:40 acetonitrile/methanol solution to the manifold and allowing the solution to enter but not leave the cartridge. The cartage soaked for five minutes before eluding into the falcon tube below for two minutes.

The samples are then dried in the TurboVap for 30 minutes at 55 degrees Celsius under air until the total volume is under 2 mL. Then the volume of the sample is adjusted to 2 mL with Honeywell LCMS grade methanol. Before the sample was placed in the mass spec vial it was syringe filtered.

LC/MS/MS Analysis

A calibration curve for 22/23 analytes was developed by running seven concentrations of PFAS standards in triplicate. The concentrations of 98 ppb, 49 ppb, 2.4 ppb, 12.25 ppb, 4.9 ppb, 0.98 ppb, 0.49 ppb were used to create the calibration curves. The limits of detection were then calculated by taking the standard deviation of the ratio of native to internal standard and then using the calibration curves to calculate the limit of detection. The highest limit of detection was 0.94 ppt for PFHxS.

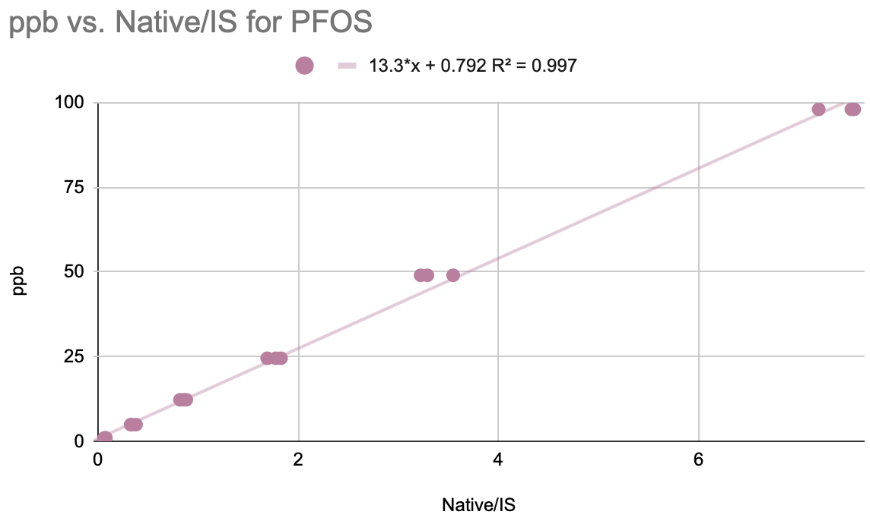
Artificial turf Experiments

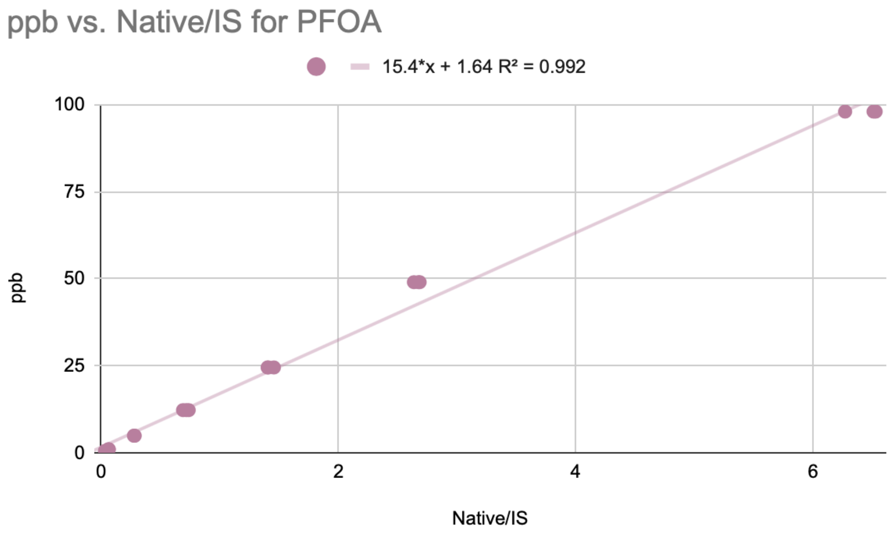
Thirteen 4x4 inch artificial turf squares were retrieved from local home improvement stores. Home Depot and Lowe’s hardware each provided two samples. The information about each sample (color, model number, ect.) was recorded before the sticker was removed from each sample. Next, 600mL of water was measured and added to each of the five HDPE bottles. In order to mimic the acidity of rainwater, 600 uL of glacial acetic acid was added to each sample to adjust the pH to 5.5. Then the artificial turf squares were placed in the water, the container was inverted three times and placed on the counter to soak for 96 hours. Once soaked, the squares were removed and the water was placed in the fridge before extraction. After the initial extraction, turf samples were dried for three days. At the end of three days, another extraction was performed using 50% acetonitrile and 50% water. This was done to change the polarity of the extraction fluid and collect lingering PFAS compounds. The PFAS internal standard was added directly to the extract (no solid phase extractions were performed on these samples).

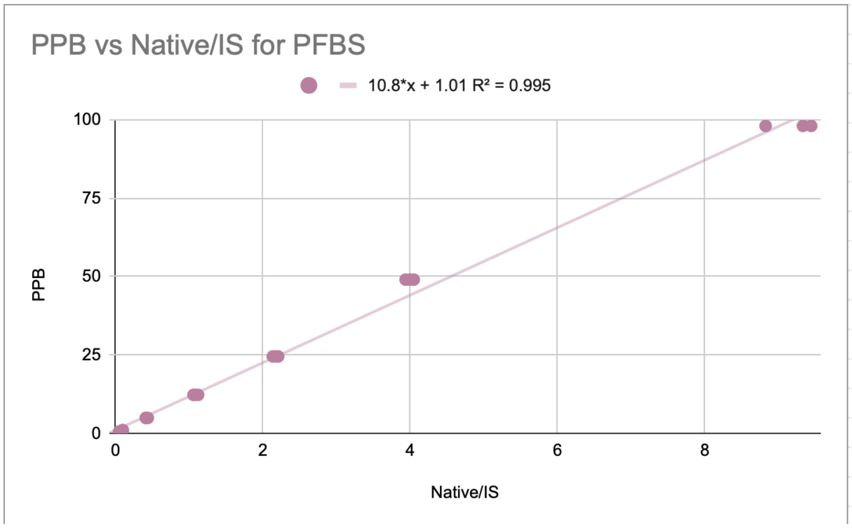
Results

Calibration curves

Calibration curves were developed for 22/23 analytes. No data was acquired for the mass labeled FOSA (Perfluorobutanesolfonoic acid) or the FOSA in the standard. N-ET-FOSAA (2-(N-Ethyl perfluorooctane sulfonamido) acetic acid) was also not detected by the instrument. We believe this is due to issues in the solid phase extraction method. In the future, literature on various solid phase extraction methods and the chemistry of these particular compounds will be searched to explain why these compounds are not present during analysis.

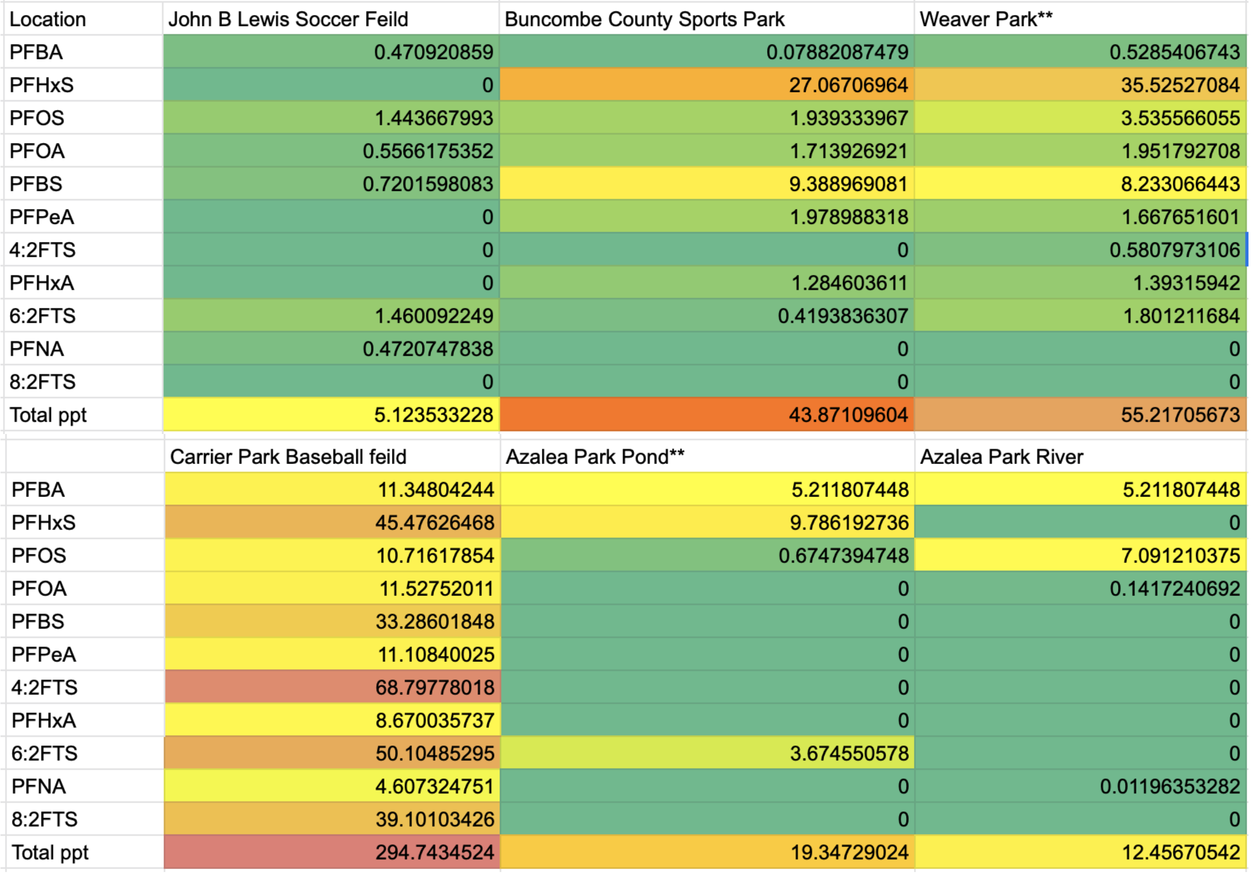

ppb vs. Native/IS for PFOS
13.3*x + 0.792 R² = 0.997
ppb
100
75
50
25
0
0
2
4
6
Native/IS


ppb vs. Native/IS for PFOA
15.4*x + 1.64 R² = 0.992
ppb
100
75
50
25
0
0
2
4
6
Native/IS



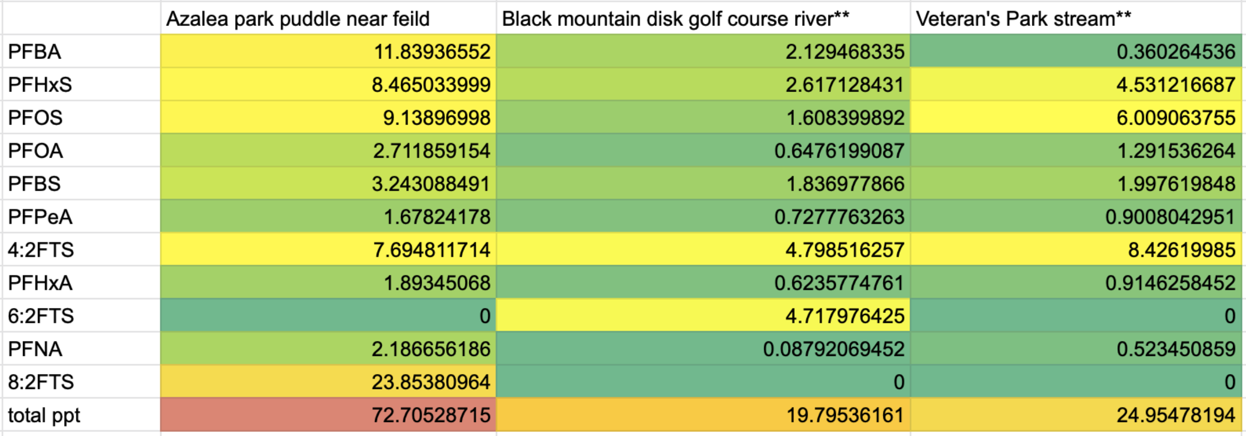
Water Samples

Twelve samples were collected from various bodies of surface water. Six of those were controls (not located near an artificial turf field. Six of these samples were located in close proximity to artificial turf fields, including one drainage puddle at the edge of a field. Charts containing select PFAS compounds found at higher concentrations are listed below. The “**” next to a location indicates that the location was not located close to an artificial turf field (control location).

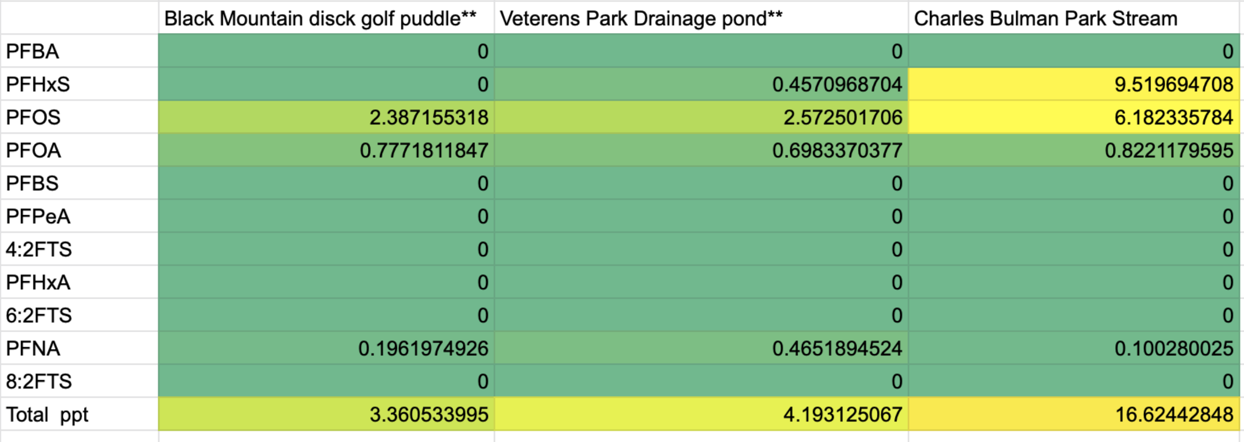


Location	John B Lewis Soccer Feild	Buncombe County Sports Park	Weaver Park**
PFBA	0.470920859	0.07882087479	0.5285406743
PFHxS	0	27.06706964	35.52527084
PFOS	1.443667993	1.939333967	3.535566055
PFOA	0.5566175352	1.713926921	1.951792708
PFBS	0.7201598083	9.388969081	8.233066443
PFPeA	0	1.978988318	1.667651601
4:2FTS	0	0	0.5807973106
PFHxA	0	1.284603611	1.39315942
6:2FTS	1.460092249	0.4193836307	1.801211684
PFNA	0.4720747838	0	0
8:2FTS	0	0	0
Total ppt	5.123533228	43.87109604	55.21705673

	Carrier Park Baseball feild	Azalea Park Pond**	Azalea Park River
PFBA	11.34804244	5.211807448	5.211807448
PFHxS	45.47626468	9.786192736	0
PFOS	10.71617854	0.6747394748	7.091210375
PFOA	11.52752011	0	0.1417240692
PFBS	33.28601848	0	0
PFPeA	11.10840025	0	0
4:2FTS	68.79778018	0	0
PFHxA	8.670035737	0	0
6:2FTS	50.10485295	3.674550578	0
PFNA	4.607324751	0	0.01196353282
8:2FTS	39.10103426	0	0
Total ppt	294.7434524	19.34729024	12.45670542



	Azalea park puddle near feild	Black mountain disk golf course river**	Veteran's Park stream**
PFBA	11.83936552	2.129468335	0.360264536
PFHxS	8.465033999	2.617128431	4.531216687
PFOS	9.13896998	1.608399892	6.009063755
PFOA	2.711859154	0.6476199087	1.291536264
PFBS	3.243088491	1.836977866	1.997619848
PFPeA	1.67824178	0.7277763263	0.9008042951
4:2FTS	7.694811714	4.798516257	8.42619985
PFHxA	1.89345068	0.6235774761	0.9146258452
6:2FTS	0	4.717976425	0
PFNA	2.186656186	0.08792069452	0.523450859
8:2FTS	23.85380964	0	0
total ppt	72.70528715	19.79536161	24.95478194

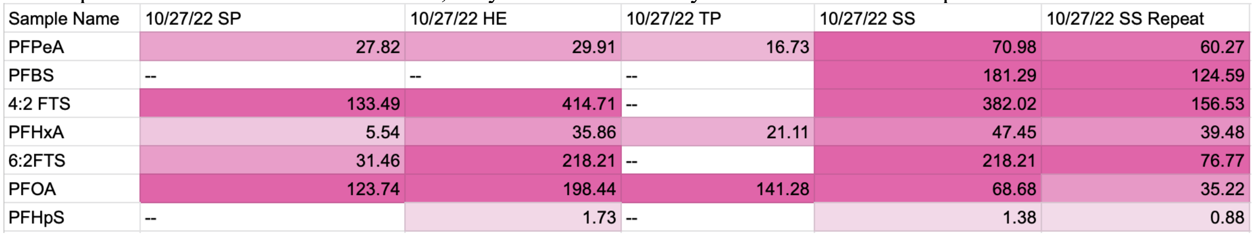


	Black Mountain disck golf puddle**	Veterens Park Drainage pond**	Charles Bulman Park Stream
PFBA	0	0	0
PFHxS	0	0.4570968704	9.519694708
PFOS	2.387155318	2.572501706	6.182335784
PFOA	0.7771811847	0.6983370377	0.8221179595
PFBS	0	0	0
PFPeA	0	0	0
4:2FTS	0	0	0
PFHxA	0	0	0
6:2FTS	0	0	0
PFNA	0.1961974926	0.4651894524	0.100280025
8:2FTS	0	0	0
Total ppt	3.360533995	4.193125067	16.62442848

The following analytes occurred at more than one location containing artificial turf at a level of over 5 ppt: 8:2FTS, 4:2FTS, PFHxS, PFBA and PFOS. These analytes were also found in the sample taken directly from a puddle at the edge of an artificial turf field (Azalea park puddle near field). Due to the voluntary disbanding of PFOS in the early 2000's, the presence of PFOS could be a result of contamination from other sources (landfills, factories, fire fighting foam runoff and other sources). The ban is voluntary, which could mean that the artificial turf is a source of PFOS contamination. Several of the control locations contained higher amounts of PFAS than expected. This could be due to unknown sources of contamination upstream.

Artificial turf Samples

The first round of extractions (deionized water and glacial acetic acid) yielded concentrations of PFAS compounds below one part per trillion. The second round of extraction sucuessfuly extracted PFAS compounds from the artificial truf. PFPeA, PFBS, 4:2 FTS, PFHxA, 6:2 FTS, PFOA and PFHpS, were all found in sample "SS", as well as the re-run of that sample. This sample contained levels of over 100 ppb of PFBS and 4:2 FTS. Sample "SP" contained levels of above 100 ppb both 4:2 FTS and PFOA. Sample "HE" contained levels of over 100 ppb of 4:2 FTS, 6:2 FTS and PFOA. Sample "TP" also contained PFOA levels of over 100 ppb. PFOA was found in all samples. Due to instrumentation error, only 10/22 PFAS analytes were unable to be quantified.



Sample Name	10/27/22 SP	10/27/22 HE	10/27/22 TP	10/27/22 SS	10/27/22 SS Repeat
PFPeA	27.82	29.91	16.73	70.98	60.27
PFBS	--	--	--	181.29	124.59
4:2 FTS	133.49	414.71	--	382.02	156.53
PFHxA	5.54	35.86	21.11	47.45	39.48
6:2FTS	31.46	218.21	--	218.21	76.77
PFOA	123.74	198.44	141.28	68.68	35.22
PFHpS	--	1.73	--	1.38	0.88

Conclusion

Of the twenty two PFAS compounds analyzed, eleven compounds were seen at levels greater than five ppt in water samples. Of those compounds, six were detected at multiple locations containing artificial turf. Those compounds were 8:2FTS, 4:2FTS, PFHxS, PFBA and PFOS. Several control sites contained more PFAS contamination than expected, leading to further questions on where this contamination could be coming from. In the acetonitrile extractions, several compounds showed up in the 100 ppb range. A possible source of PFAS contamination is artificial turf. PFBA, 4:2 FTS, PFOA, PFPeA and 6:2 FTS are the most likely compounds to come off of artificial turf.

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