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High Resolution Per and Polyfluoroalkyl Substance Mass Balance in a Wastewater Treatment Plant Post Aqueous Film Forming Foam Spill

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<u>Abstract</u>

First-generation, PFOS-dominant Aqueous Film Forming Foam (AFFF) concentrate of approximately 200 gallons was released to the sewer system from the Kalamazoo/Battle Creek International Airport on March 30, 2021. The AFFF release migrated to the Kalamazoo Water Reclamation Plant (KWRP), raising plant effluent above the Department of Environment, Great Lakes, and Energy (EGLE) PFOS water quality standard for a total of 46 days post-spill, although the influent concentrations exhibited sharp declines after only 7 days. By sampling influent, effluent, and biosolids daily, the only known long duration, high-frequency PFAS data set of an accidental AFFF release was generated. This study has four primary objectives: (i) identify AFFF released from the airport by comparing influent PFAS composition ratios to those of AFFF from different manufacturers; (ii) perform a plant-wide mass balance to discern how PFAS, and in particular PFOS, moves through and leaves the plant; (iii) provide constraints on the mass balance by estimating the aqueous volume of the AFFF release; and (iv) determine plant mechanisms responsible for 46-day PFOS exceedance of EGLE water quality standard in effluent. This study is the first of its kind to show that a mass balance for an accidental PFAS release is feasible given sufficient sampling resolution and laboratory consistency. Results from the mass balance indicate 1227 grams of PFOS entered the plant as influent, 704 grams of PFOS (54% of total output) discharged into the Kalamazoo River as effluent, and 610 grams of PFOS (46% of total output) sorbed to biosolids and landfilled. PFOS output mass is 84 grams (7%) higher than input PFOS mass; this apparent discrepancy may indicate PFOS generation within the KWRP due to chemical transformations of precursors. Identification of the AFFF formulation and spill volume constrained the mass balance and increase the confidence of the study results. Recirculatory and desorption processes are likely responsible for the extended exceedance of the EGLE water quality standard. These findings provide critical insight to wastewater treatment plant operators worldwide for performing PFAS mass balances and developing operational procedures for accidental PFAS releases that minimize PFAS mass releases to the environment.

Introduction

Per- and Polyfluoroalkyl Substances (PFAS) are a group of manufactured chemicals originally developed in the 1940s. Now, over eight decades later, PFAS have become the center of public focus due to their mobility, high resistance to degradation, and known adverse health impacts (Brase et al., 2021). PFAS are used in a wide variety of household products and industrial processes. PFAS are used in nonstick pans, water-resistant clothing, grease-resistant paper, food wrappers, and cosmetics. Industrially, PFAS have been used in aqueous film-forming foams (AFFF), textile manufacturing, paper production, metal coating, tanneries, and many other applications. Sources of PFAS to the environment include atmospheric emissions and deposition, landfills receiving PFAS-laden waste, on-site contamination from PFAS-using industries, accidental PFAS spills, and wastewater treatment plants (WWTP) (Helmer et al., 2021). These sources lead to contamination of soil, groundwater, surface water, drinking water, and aquatic and terrestrial ecosystems.

WWTPs passively receive PFAS from wastewater streams originating from landfill, local industries, and municipalities. WWTPs are not designed to treat PFAS (Lester & Birkett, 2019), and collectively, effluent discharge from WWTPs serves as the greatest aqueous release mechanism of PFAS to the environment (Helmer et al., 2021). Consequently, PFAS discharges to WWTPs are an area of high research interest (Cookson & Detwiler, 2022). A generalized description of wastewater movement within WWTPs is as follows: water enters the WWTP as untreated influent that first undergoes primary treatment via physical settling, moves on to secondary biological/chemical treatment, and lastly undergoes tertiary polishing/disinfection prior to discharge. During the primary and secondary treatment stages, solid residuals are separated from the aqueous waste stream. Liquid effluent is typically released to receiving waterways, and solid residuals (biosolids/sludge) are either landfilled or applied to agricultural fields. The secondary biological/chemical stage accounts for the majority of wastewater treatment by utilizing microbes under both oxidizing and reducing conditions to degrade biological matter, contaminants, and nutrients. Central to biological treatment is the management of microbes (often referred to as "bugs") that are continuously recirculated within the plant to optimize degradation. The

recirculation of microbes leads to a distribution in the duration that activated sludge remains in the plant. The solids residence time (SRT) is a common metric computed by WWTP operators that describes the age of the sludge.

Concentrated PFAS releases to WWTPs are in all likelihood fairly common, yet largely go unreported and undetected due to the lack of environmental regulations necessitating compliance reporting and sampling. Examples of known AFFF spills to WWTPs in Michigan include the Menominee Warehouse Fire in October 2022 which involved extinguishing a large-scale industrial fire at a paper plant using AFFF. The AFFF and water mix were mostly contained and later sent to the Menominee WWTP, where the PFAS-laden effluent was then discharged to the Menominee River (Miller, 2022). Another example occurred at the Wurtsmith Air Force Base in Oscoda, Michigan where AFFF releases within the base occurred during fire suppression training activities from the early 1980s to 1992. In 1985, three water wells were installed 500 yards downgradient from where AFFF used in fire suppression training routinely infiltrated base soil. These wells provided PFAS-laden water to the base and ultimately to a nearby WWTP that discharges effluent to the adjacent Clark's Marsh and Ausable River (Minor, 2020).

PFAS releases to WWTPs are nonexistent in the literature, except for a single study involving a simulated AFFF release to a WWTP (Gonzalez et al., 2021). The study used sequencing batch reactors to simulate a WWTP, added a fluorotelomer sulfonate (FTS)-based AFFF to the system, and estimated that 70% of the AFFF would exit the plant within four days in the form of effluent and solids. Applications of these findings are limited as the simulated system did not include the solid retention times and recirculatory processes characteristic of most modern WWTPs.

On March 30, 2021, at 10:00 am, AFFF concentrate of approximately 200 gallons was released from the Kalamazoo/Battle Creek International Airport. The spill occurred when a worker, attempting to empty the water storage tank of an Oshkosh T-1500 fire truck, opened the valve to the AFFF concentrate tank by accident. The water tank valve was then immediately opened afterwards, and the AFFF was flushed into a drain connected to the Kalamazoo sewer by approximately 1300 gallons of water. The

AFFF migrated through the sewer network to the Kalamazoo Water Reclamation Plant (KWRP) (Figure

1), with initial detection occurring four hours after the release. Upon notification of the spill, KWRP sampled plant influent, effluent, and biosolids using knowledge of sewer pathways and transport times. Samples were tested using a State of Michigan mandated 28-analyte PFAS suite with concentrations reported in ng/L (equivalent to parts-per-trillion (ppt)) for aqueous samples or ng/g (equivalent to parts-per-billion (ppb) for biosolid samples). Upon receiving initial sample results (five days after they were taken due to a rush request, normal laboratory turnaround times are on the order of 3-4 weeks), plant influent, effluent, and biosolids exhibited elevated PFAS concentrations, particularly PFOS. Near daily samples were then collected for all three components (influent, effluent, and biosolids) until



Figure 1: Sewer line map from Kalamazoo/Battle Creek International Airport to KWRP. Map courtesy of the City of Kalamazoo.

plant effluent declined below the Department of Environment, Great Lakes, and Energy (EGLE) PFOS water quality standard of 12 ng/L. Airport surfaces and sewer lines were cleaned on May 3 through May 7, 2021, to flush residual PFAS in the sewer line, and these activities generated a secondary PFAS spike in influent concentrations to KWRP.

This AFFF spill and the near-daily sampling that followed provide an excellent opportunity for an in-depth study of a concentrated PFAS release to a WWTP. PFAS analysis is expensive (~\$300 per sample) and costs typically preclude daily sampling within WWTPs. However, given the exceedance in water quality standards, influent, effluent, and biosolids were sampled 46, 67 and 49 times, respectively,

generating a long duration, high-resolution dataset with an analytical cost of \$182,000. Combined, this dataset is quite unique given the continuous nature of sample collection and is ideal for studying temporal trends and the fate and transport of PFAS within a modern WWTP.

This study has four main objectives: (1) Identify AFFF formulation released from the airport by comparing influent PFAS composition ratios to those of AFFF from different manufacturers; (2) Perform a plant-wide mass balance to discern how PFAS, and in particular PFOS, moves through and leaves the plant; (3) Provide constraints on the mass balance by estimating the amount of the AFFF release; and (4) Determine WWTP mechanisms responsible for observed PFOS exceedance of EGLE water quality standard in effluent for 46 days post-AFFF-release.

Methodology: Mass Balance Approach

Isolating Spill Signal

A PFAS background correction was performed to isolate the AFFF spill signal. The KWRP receives PFAS from various sources, including landfill leachate and industrial and municipal wastewater. PFAS concentrations for influent, effluent, and biosolids are comprised of 12 influent samples from May 4 to October 13, 2020, 7 effluent samples from September 21 to October 1, 2020, and five biosolid

samples from March 25 to March 29, 2021. Average background concentrations are then subtracted from all the respective influent, effluent, and biosolids post-spill concentrations. Isolated influent, effluent, and biosolids concentrations plotted on a logarithmic axis versus time after the spill are shown in Figure 2.



Figure 2: PFOS concentrations vs days after spill.

Mass Balance

The mass balance approach is used to quantify PFAS entering and exiting the WWTP and the distribution of PFAS mass across various components within the WWTP. This is performed on a plant-wide scale for the AFFF release. Conceptually simple, wastewater influent enters the plant as an input and treated effluent and biosolids exit the plant as outputs:

$$M_i = M_e + M_b \pm \Delta M,\tag{1}$$

where M_i is influent PFOS mass, M_e is effluent PFOS mass, M_b is biosolid PFOS mass, and ΔM is the difference of PFOS mass entering versus leaving the WWTP. This mass balance evaluates the current data to assess if sufficient data resolution is present to perform a plant-wide PFAS mass balance with reasonable error constraints on individual components. Influent and effluent sample concentrations are converted to daily mass as a product of concentration and volumetric flow rate:

$$M = CQ,$$
 (2)

where M is PFOS mass in nanograms, C is PFOS concentration in nanograms per liter, and Q is daily volumetric flow of the wastewater stream in liters. Mass of PFOS in biosolids is computed according to:

$$M_b = C_b P M_T, (3)$$

where M_b is PFOS mass in nanograms, C_b is dry weight PFOS concentration in nanograms per gram, M_T is mass in grams of centrifuged biosolids, also known as cake, hauled out of the WWTP, and P is percent total solid of the centrifuged biosolids.

The PFOS mass balance was conducted from the day of the spill (day 0) to 50 days afterward and used sample data that was supplemented as needed with interpolation (influent, effluent, biosolids) between known samples and extrapolation (biosolids) into the future. Estimating the initial slug is particularly challenging given the poor sampling resolution at the beginning of the release. Only two samples were taken on the first day (day 0 for influent and day 1 for effluent due to 24-hour retention time between incoming influent and outgoing effluent) and no samples were collected over the next 5 days until laboratory results indicating elevated PFAS were available. Sewer travel times from

Kalamazoo/Battle Creek International Airport to the KWRP are on average four hours. Utilizing the known transport time range, KWRP operators collected samples four and six hours after the AFFF spill. Several options of computing the original slug were considered; however, a simple piston of the average of the first two concentrations was applied over a six-hour flow period. This simple approach was ultimately adopted as it honors the observed initial high concentrations over the distribution of known travel times in the sewer lines and accounts for uncertainty in mass arrival times attributed to fluctuating sewer flux rates and pipe dispersion.

Best-fit power-law trendlines to concentration data from the first 20 days (R² equal to 0.89 and 0.99 for influent and effluent, respectively) are used to compute missing concentration data for days 1-4 (influent) and 2-5 (effluent). Day 6 for influent was not sampled and instead was interpolated using an average of days 4 and 6. Extrapolating biosolid concentrations to match influent and effluent sample periods is performed by fitting an exponential trendline to the biosolid concentration data. The initial peak, which deviated from the later-time trend, is excluded when fitting the trendline equation for days 41 to 50 after the spill. The data indicate a strong exponential trend with an R² of 0.97. The regression equation was then used to extrapolate biosolids concentrations for days 41-50. Once influent, effluent, and biosolid concentrations are estimated (either directly from samples, interpolated, or extrapolated), equations (2) and (3) are used along with daily WWTP flow rates, total cake hauled, and percent total solids. These data were provided by KWRP operators.

Mass Balance Constraints

Identifying the spilled AFFF formulation involved analysis of plant influent PFAS compositions and concentrations. The spill report identified the AFFF concentrate as Ansulite AFC-3A 3% with an accompanying safety data sheet showing a second-generation fluorotelomer-based formulation. The KWRP sample data, however, unequivocally show that the AFFF release is a first-generation PFOSdominant formulation. To identify the AFFF formulation that was spilled, various AFFF PFAS samples were obtained from the Michigan PFAS Action Response Team (MPART) AFFF records. Concentration ratios for PFAS compounds in various formulations were then calculated and compared to the respective ratios in plant influent.

The volume of AFFF release is estimated using:

$$S = \frac{M}{R}, \qquad (4)$$

where *S* is the volume of AFFF concentrate (gal), *M* is PFOS mass computed in the KWRP influent (kg), and *R* is the ratio of PFOS per gallon of the AFFF (kg/gal) for the best match to the MPART AFFF data. Note that spill involved AFFF concentrate and necessitated rescaling from 6% (i.e., 6 parts concentrate to 94 parts water) in the MPART data to concentrate form.



estimated inputs and outputs, 1227 grams entered the KWRP, and 1314 grams exited the system; effluent and biosolid PFOS mass are 84 grams or 7% higher than influent mass. The calculations indicate that 54% of PFOS exited as effluent into the Kalamazoo River, and 46% of PFOS sorbed to biosolids and was landfilled.

Results and Discussion

Comparing effluent and biosolid mass estimates, effluent is more tightly constrained as approximately 70% of effluent mass is computed from known concentration data. Biosolids, on the other hand, are likely underestimated due to reliance on laboratory desorption/extraction methods used to quantify PFAS from collected samples. While errors in the mass balance estimates for each component are unknown, it is likely that the extra PFOS mass leaving the system cannot solely be attributed to error and arises from PFAS transformations in the KWRP. The 28-analyte PFAS suite used for sampling did not detect elevated concentrations of PFOSA, MeFOSAA, or EtFOSAA in the plant influent which are known PFOS precursors. Recent studies suggest that other undetected PFOS precursors are commonly present in first-generation AFFF formulations. For example, the electrochemical fluorination process responsible for creating the PFOS-dominant AFFF does not lead to a clean chemical synthesis, and often generates unknown and unwanted byproducts (Berger et al., 2011). These undetected byproducts can function as precursors required for PFOS transformation and production, as illustrated by Choi et al. (2022). Gonzalez et al. (2021) showed similar results for a fluorotelomer-based AFFF where a higher concentration of 6:2 FTS was present in effluent than influent suggesting one or more precursor PFAS components were transformed to 6:2 FTS within a simulated WWTP.

Constraints and Spill Volume Estimate

The EGLE Ansulite AFC-3 6% AFFF concentrate best matches the spill PFAS signature received by the KWRP. Figure 4 illustrates concentrations of the five dominant PFAS compounds in the AFFF and influent samples (summing to approximately 91% of total PFAS)



Figure 4: Radar plot comparing Ansulite AFFF formulation and plant influent.

on a radar plot, and demonstrates the utility of identifying AFFF formulations based on plant influent concentrations. Figure 4 also reiterates PFOS as the dominant PFAS compound and illustrates why it is

the focus of this study. Additionally, the strong match with the Ansulite AFFF formulation provides confidence in our background corrections used to isolate the spill signal, and more importantly, constrains total PFAS mass for the spill.

The spilled AFFF amount is calculated from influent PFOS mass and combined effluent plus biosolid mass. Calculated values are 223 and 238-gallon releases, respectively. The AFFF concentrate tank on an Oshkosh T-1500 is 200 gallons meaning the spill amount calculated from influent PFOS mass is 12% higher than expected, and the spill amount calculated from combined effluent and biosolid PFOS mass is 19% higher than expected. The high value for spill amount from effluent and biosolid PFOS mass could partially result from PFOS transformations. The 12% overestimation from influent PFOS mass is likely the result of using one measurement to represent PFOS concentrations for a 24 hour period, unaccounted PFOS located in the airport storm drain from previous spills and other analytical errors discussed later.

Recirculation

the aeration tanks), is

The mechanism likely responsible for the 46-day exceedance of EGLE PFOS water quality standard in effluent post-AFFF release is plant recirculation via solid retention time (SRT). To calculate SRT, total pounds of Total Suspended Solids (TSS) in the entire secondary system (including the final clarifiers and

normalized by the daily TSS pounds wasted in waste mixed liquor plus the TSS in the effluent. In other words, values of SRT are characteristic of plant recirculation. Effluent PFOS concentrations over time exhibit exponential decay



Figure 3: PFOS concentration vs time after spill

EFfluent PFOS & PFHxS Concentration vs Time

(Figure 5). The best-fit exponential decay equation to these data yields a half-life of 5.8 days and a fulllife of 8.3 days. Compared to the KWRP data, the calculated full-life is 73% of the SRT. Further research is required to understand the relationship between PFOS decay and recirculatory processes fully. Still, the value of this analysis is three-fold. First, the data demonstrates an exponential relationship between PFOS concentration and time after spill. Second, it shows that SRT (a standard metric computed for all WWTPs) may be related to the rate at which PFOS, and presumably other PFAS, decays within WWTPs. Third, this analysis suggests that modifying plant operations controlling recirculation processes will alter the rate or efficiency at which PFOS decays in the effluent stream. This idea is supported by Kibambe et al.) (2020) where longer SRT leads to greater mixed liquor suspended solid concentrations and PFAS removal. Additionally, Arvaniti & Stasinakis (2015) suggests that powdered activated carbon is more effective than granular activated carbon, and anion exchange resin has a high removal efficiency for PFOS and PFOA.

Sources of Error and Study Limitations

There are many sources of error in conducting plant-scale PFAS mass balances from an accidental sewer release. The first source of error comes from the frequency of sampling. Despite sampling on a near-daily basis, one instantaneous sample is unlikely to capture the variability in concentration given temporal trends in PFAS concentrations and external factors that control influent flow rates to the plant. The infrequency of sampling during the initial slug represents another source of error. The use of only two points to represent the initial concentration peak is likely the largest source of error in the mass balance. Additionally, missing data points and their associated interpolations or extrapolations could have introduced error. Lastly, the mass balance equations for influent and effluent require concentrations to be multiplied by flow rate. Flowmeters used to track the amount of influent and effluent moving through the plant have accuracies of +/- .1 million gallons per day. When multiplied by concentration, this error could compound by a larger degree. Despite these sources of error, most of which are common for all WWTPs, the estimated spill volume of AFFF (and equivalent PFOS mass)

served as a valuable constraint on total PFAS mass received by the plant. PFOS mass computed in the plant influent indicates a 12% overestimation of AFFF concentrate. This overestimation may be attributed to a combination of the error sources and residual AFFF in the airport drain from previous spills. A 7% greater mass in the effluent PFOS than influent could reflect mass balance error as well as precursor transformation to PFOS within the plant.

Conclusion

This is the first study to demonstrate a mass balance for an accidental PFAS release can be performed given sufficient sampling and laboratory consistency. Results from this study show that 1227 grams of PFOS entered the plant as influent, 704 grams of PFOS (54%) were released as effluent into Kalamazoo River, and 610 grams of PFOS (46%) were sorbed to biosolids and landfilled. Output PFOS mass was 84 grams (7%) higher than input PFOS mass; this increase in PFOS may indicate PFOS transformations in the WWTP. Identifying AFFF formulation and spill amount constrained mass balance components and added confidence to the mass balance approach implemented in this study. Lastly, this study was able to illustrate a potential link between recirculatory processes and extended exceedance of the EGLE water quality standard for PFOS.

In the case of future spills, the several suggestions are recommended to ensure mass balance accuracy. Collect samples more frequently (hour scale) or take 24-hr composites during the period at which the initial spill slug is estimated to reach the WWTP. Do not wait for initial sample results to see if there is elevated PFAS; instead, assume PFAS levels are elevated and continue sampling on a sub-daily basis until initial laboratory data are provided. Sewer cleaning plans should be formulated for potential spill sites to flush out residual PFAS. Study data showed that PFOS concentrations continued to be elevated until sewer cleaning occurred. The duration of elevated concentrations is likely due to pipe dispersion and sorption to sediments and solids within the sewer distribution system.

Future research is required to determine how long sewer cleaning should occur after the spill. The fastest way to decrease PFOS levels entering the WWTP would have been to clean the sewer systems

as soon as possible. This, however, could result in adverse ecological effects due to higher rates of PFOS loading to the environment in the initial time period following the spill due to lower rates of biosolids/sludge sorption. A joint optimization of recirculation processes and addition of powdered activated carbon would be advantageous to identify the most effective way to remove PFAS from accidental WWTP releases.

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