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PFAS reduction during biosolids drying correlates to initial moisture content and is accompanied by detection of PFAS in dryer condensate

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Land application of biosolids has come under scrutiny for its potential to convey PFAS. Some water resource recovery facilities (WRRFs) have been forced to choose between implementing more extensive biosolids treatment technologies to achieve regulatory limits or altering their biosolids handling approach. Many novel biosolids treatments for PFAS removal include advanced thermal processes such as pyrolysis or gasification. Such processes require drying of biosolids as a preparatory step for treatment. However, little is known about the isolated impact of biosolids drying on the removal of PFAS from biosolids, or what fate PFAS may undergo during the drying process. The objective of this research was to elucidate how biosolids' PFAS profiles change due to drying, and to understand if PFAS can be transported with the gaseous vapor phase into liquid condensate. Biosolids were collected from five WRRFs and dried in a lab-scale oven. Additionally, biosolids from one of the WRRFs were dried in a system that captured and condensed vapor in a methanol impinger. Analysis of the PFAS profiles of the biosolids before and after drying indicated an average of 58% reduction in detectable PFAS across all sites, with the range being 43–74%. A positive correlation was established between the initial moisture content of the biosolids and reduction in detectable PFAS (R^2 = 0.61). The condensate contained 12–22% of the initial PFAS from the wet biosolids, indicating that PFAS can leave with the gas-phase vapor generated during drying. This research supports the conclusion that biosolids drying can reduce detectable concentrations of PFAS, and operational parameters could be adjusted to optimize dryers for this purpose. Treatment technologies could then be applied to the condensate to destroy the PFAS contained therein.

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Water impact

Reduction of PFAS concentrations in biosolids may be necessary in some regions to meet land application regulations. While the implementation of novel treatment technologies may be prohibitive in terms of scale and energy costs, biosolids drying is a conventional technology that can reduce the concentration of PFAS in biosolids. After drying, some PFAS were detected in dryer condensate, indicating that treatment of condensate could disrupt PFAS cycles within WRRFs.

1.0 Introduction

Per- and polyfluoroalkyl substances (PFAS) are a recalcitrant group of chemicals that are ubiquitous throughout society and have been associated with several human health concerns, including decreased antibody response, decreased fetal growth, increased risk of kidney cancer, and neurotoxicity.[1–4] Due to their presence in consumer goods and industrial discharges, many PFAS end up in wastewater and biosolids.[5–7] Land application of biosolids has been scrutinized recently for its capacity to convey PFAS into the environment.[7,8] As a result, some states have restricted land application based on the PFAS concentration in biosolids, and other states have banned it entirely.[8,9] Now, water resource recovery facilities (WRRFs) must account for PFAS when designing their biosolids management plans.

Land application has long been regarded as one of the most cost effective biosolids handling methods, as well as a sustainable method for recovering nutrients.[10–12] Employing

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land application to manage biosolids is also beneficial for reducing the amount of material sent to landfills.[10] Landfills emit volatile PFAS to the atmosphere and have potential to leach PFAS into nearby groundwater.[13,14] Moreover, sending biosolids to landfills may become costly or impracticable as space becomes limited and moisture content becomes a concern. Alternatively, incinerators, though potentially capable of defluorinating PFAS, may generate toxic gases like dioxin, furan, and fluorocarbons, including short-chain PFAS.[6,15,16] Furthermore, new incinerators are difficult to install if not part of an existing incinerator upgrade or expansion. Thermal processes have garnered recent interest as possible solids handling processes that could help mitigate PFAS in biosolids. Pyrolysis has been shown to reduce the detectable concentrations of PFAS in biochar, and gasification also likely reduces PFAS in the solid-phase.[17–20] Drawbacks to these emerging thermal processes include that they alter the inherent value of biosolids because biochar does not contain the same available carbon and nutrient content, and the long-term operational feasibility of these processes is still unknown.[21–23] Implementation of these thermal processes would require drying of the feed biosolids.[20,24,25]

Interestingly, drying may provide an avenue to help manage PFAS in biosolids. Previous research found that detectable PFAS concentrations were lower in biosolids after drying, in both a lab-scale oven and a full-scale dryer, and that reduction of detectable PFAS concentration was positively correlated to the initial moisture content of the biosolids sample.[26] Other research investigating the phenomenon of PFAS reduction during biosolids drying is limited, and comparison of data is complicated by varying analytical techniques. For example, other studies found PFAS concentrations increased during biosolids drying but analyzed fewer species.[27–29] The study which analyzed the most distinct PFAS species saw both increases and decreases to the PFAS profile of biosolids during drying across multiple sites.[30]

While multiple studies have reported decreases in detectable PFAS concentration, little information is available that elaborates on the mechanism by which this occurs. Only one study has captured and analyzed the vapor from the drying process, and in this full-scale study the mass of PFAS entering the regenerative thermal oxidizer (RTO) from a rotary biosolids dryer was orders of magnitude lower than the mass of PFAS removed from biosolids during drying.[31] More research is needed to determine the removal mechanism and phase distribution of PFAS during and after biosolids drying. Reductions in PFAS concentration have been observed at temperatures of 105 °C, which excludes mineralization as a removal mechanism; therefore, it is plausible that PFAS are being transferred to the vapor generated during drying.[63] Understanding the transport and fate of PFAS during biosolids drying will reveal avenues for appropriate management, including possible treatment, of PFAS after drying.

The objective of this research was to determine the reduction of detectable PFAS during biosolids drying across a range of moisture contents in biosolids and to establish if biosolids drying yielded PFAS in the gas-phase that could be detected in downstream condensate. It was hypothesized that higher moisture content would yield greater reductions in detectable PFAS concentration and that PFAS would be removed from biosolids during drying *via* aerosolization and appear in the captured condensate.[26,32,33] To test these hypotheses, triplicate samples of biosolids from five full-scale WRRFs were collected and dried in a lab-scale oven. The vapor from one set of drying experiments was captured with a methanol impinger to measure PFAS in condensate. This work sought to elucidate the feasibility of PFAS removal *via* transport with water during biosolids drying and determine the fraction of detectable PFAS reduction that could be explained by transfer to condensate.

2.0 Methods

2.1 Experimental setup

Biosolids samples were collected from five full-scale WRRFs with average flowrates ranging from 30 to 200 million gallons per day (MGD). Four of the five WRRFs employ secondary treatment systems with biological nutrient removal (BNR), while one facility (WRRF no. 1) utilizes secondary treatment for biochemical oxygen demand (BOD) removal only. All WRRFs use gravity thickening followed by anaerobic digestion and dewatering with centrifuges. The biosolids from these five WRRFs were dried in triplicate in a lab-scale oven overnight (24 hours) at 105 °C to determine the reduction of detectable PFAS achieved during biosolids drying. Biosolids from five different WRRFs were used for experiments to better understand how varied initial moisture contents impact PFAS removal. For these drying experiments, the wet and dry biosolids samples (before and after drying, respectively) were analyzed for PFAS as described below.

In addition to the oven-drying experiments, one set of biosoilds was dried in triplicate in a lab-scale heating system that allowed for vapor collection. The vapor collection system was customized by modifying a high-temperature conversion system (SI Fig. S1).[34] The customized system included a stainless-steel reactor vessel, a ceramic radiative heater, an inert gas purge system, and a gas collection system consisting of one impinger (20 mL of methanol, HPLC grade, >99.8%, Alfa Aesar™) in an ice bath. A pressure gauge and a thermocouple were used to monitor pressure and temperature inside of the reactor, respectively. The temperature of the system was raised at a rate of 8–15 °C min^{-1} to 105 °C, and the residence time of drying the biosolids in the reactor was 12 hours. To capture the system's off-gas, vapor from one drying experiment passed through PFAS-free tubing to the methanol impinger, where it was condensed (and henceforth referred to as condensate). The mass of the dried biosolids was determined gravimetrically, and the mass of the PFAS in the condensate was determined

as a function of their concentration in the methanol impinger and the methanol + water volume; the volume of liquid in the impinger increased during the experiments from the water that transferred from the wet biosolids to the impinger during drying. The initial wet biosolids and the liquid from the impinger were stored in a freezer until shipment to Eurofins. The dry biosolids were stored at room temperature until shipment to Eurofins.

Negative controls were run to check for background PFAS contamination in this gas-phase collection system. Baked Ottawa sand (20–30 mesh, Spectrum™ Chemical) was loaded into the reactor, and 20 mL of methanol were loaded into the impinger. No detects were reported in the sand. The only species that was detected in the methanol negative controls was perfluoropropionic acid (PFPrA), at 1.4 μg L^{-1} in the methanol impinger. PFPrA was excluded from analysis for all gas-phase methanol samples because no samples had PFPrA concentrations greater than 10× the blank concentration. Pure methanol did not contain any reportable PFAS. The biosolids and liquid samples were put into containers provided by Eurofins for PFAS analysis. Samples were shipped in a cooler with ice packs to Eurofins for PFAS analysis.

2.2 PFAS analysis

The PFAS analysis employed in this experiment has been outlined previously.[26] A list of the sixty targeted PFAS analytes is included in the SI, section S1. Isotopically labeled analogs, referred to as extracted internal standards (EIS), were used for quantition where available. The list of EISs and their corresponding target analytes are listed in the SI Table S1. PFAS were analyzed with a SCIEX 5500+ triple quad LC/MS/MS system operated in multiple reaction monitoring (MRM) mode. Biosolids samples were manually homogenized in their collection containers before being subsampled (nominally 1 g for pre-drying and post-drying samples). The EISs of the native analytes were spiked into each subsample, then were sonicated with basic methanol for an hour. Water was added to the extracts to re-constitute them, and then they were cleaned up *via* solid-phase extraction (SPE) using a WAX SPE cartridge (Strata X-AW or equivalent) to retain the desired PFAS. Then, basic methanol was used to elute the IDAs and target analytes from the SPE cartridge and $^{13}C_2$-PFOA was added as the internal standard (ISTD) to fortify the extracts. A solution of 80 : 20 methanol/water was added to obtain a final extract volume of 10 mL. The same extraction procedure was used for pre-drying and post-drying biosolids samples.

To ensure the accuracy of the measurements of the 60 target analytes, initial and continual verification of accuracy and precision are achieved *via* laboratory control sample aliquots, laboratory control sample duplicates, laboratory method blanks, method detection limit (MDL) studies, MDL verification aliquots, and performance testing studies. The descriptions of these processes can be found in the corresponding laboratory standard operating procedures (SOPs). These procedures and performance test results are audited *via* 3rd party laboratory accreditation bodies for the relevant regulatory programs, including the National Environmental Laboratory Accreditation Program and the United States Department of Defense Environmental Laboratory Accreditation Program.

Additional measures of method performance monitored within each sample include EIS recovery, ISTD response, analyte retention times, analyte ion ratios, and analyte signal-to-noise ratios. The criteria for these sample-specific quality controls and identification elements are specified in the corresponding laboratory SOP. PFAS concentrations are reported on the basis of a sample's dry weight, which is determined after moisture has been removed at 105 °C and the sample has cooled to room temperature in a desiccator.

2.3 Statistical analysis

Concentrations of PFAS were normalized to μmol of F per kg of dry solids, where F represents the fluorine content of a given species of PFAS. Reported data were in units of μg PFAS per kg dried biosolids, so each measurement was divided by the molecular weight of the species and then multiplied by its F content per molecule. All data are available in the SI in units of μmol of F per kg of dry solids and also as the more commonly reported values of μg PFAS per kg of dry solids. For the condensate capture and mass balance experiment, the concentrations of PFAS in the biosolids and methanol were multiplied by the sample mass or volume, respectively. As such, the amount of total PFAS was normalized to moles of F in each targeted PFAS. Biosolids from source 5 were chosen by the anonymous utility partner for this phase of the experiment.

Effective statistical analysis required complete triplicate data, therefore, non-detects in the data were filled in with the reporting limits of the analytical method where triplicate data were incomplete. By contrast, if a species was not detected across any triplicates for either wet or dry biosolids, reporting limits were not used to fill in the data. Though this approach compensates for the potential trace presence of PFAS that evades detection, it slightly reduced the calculated percent removals of total PFAS compared to not using reporting limits (values of zero). Figure captions indicate when reporting limits are included in a given figure.

Additionally, significance was set to an alpha value of 0.05 or less for paired Student's *t*-tests. All statistical analyses were conducted in GraphPad Prism v10.4.2 (GraphPad Software, La Jolla, CA).

3.0 Results & discussion

3.1 Detectable PFAS removal during drying

Drying substantially reduced the presence of detectable PFAS in biosolids. In all five biosolids sample sets the total sum of detectable PFAS concentrations was significantly lower after drying (*p*-values of 0.0021, 0.0091, 0.0012, 0.0034, 0.0023, respectively) (Fig. 1). When categorized by speciation, nearly

all classes of PFAS exhibited a measured concentration reduction during biosolids drying across all sites, including terminal species such as perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs). The average reduction in total detectable PFAS concentration across all samples was approximately 60% (57.6%), though reported reductions in concentration ranged from 42.3% to nearly 74.2% (Fig. 1). This range of percentages suggests that characteristic differences in biosolids samples impact PFAS concentration reduction during biosolids drying.

The presence of water affects the reduction in detectable PFAS during biosolids drying. The percent reduction was positively correlated to initial moisture content (Fig. 2). The R^2 value was 0.61 for a sample size of 18, which implies that 61% of variation in PFAS concentration reduction was explained by moisture content. The initial moisture contents of all samples were within a narrow range, from 72.4% (27.6% total solids) to 78.5% (21.5% total solids). This strong trendline justifies future research into a wider range of initial moisture (and total solids) contents. Also, while these data imply a mechanism of PFAS loss *via* transport with water, the exact removal mechanism cannot be determined from these data alone. The temperature of the lab-scale oven was slightly above the boiling point of water (105 °C). Previous research on the volatilization of pure chemical PFAS revealed that, at temperatures around 100 °C, thermogravimetrically analyzed weight loss of long-chain PFCAs such as PFOA, PFNA, and PFDA was less than 10%, and for PFSAs such as PFBS, PFHxS, and PFOS, weight loss was negligible.[36] The lack of removal

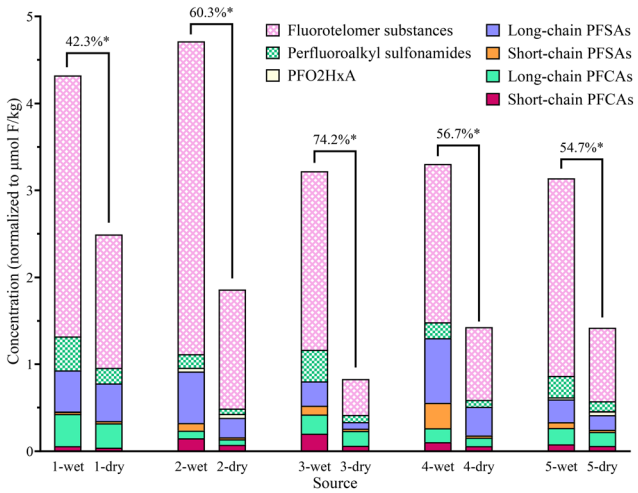


Fig. 1 Average concentration of categorized PFAS (normalized to µmol F kg^{-1}) in pre-drying ("wet") and post-drying ("dry") solids at five sites, labeled 1–5 (n = 3). Fluorotelomer substances include FTSes and FTCAs. Perfluoroalkyl sulfonamides include NMeFOSAA, NEtFOSAA, NMeFOSE, and NEtFOSE. According to the EPA, long-chain PFSAs have 6+ carbons, short-chain PFSAs have <6 carbons, long-chain PFCAs have 8+ carbons, and short-chain PFCAs have <8 carbons.[35] Reporting limits were substituted in for non-detects. Total PFAS percent removals were calculated for each triplicate and averaged (shown above each sample). Asterisk denotes statistical significance. Reprinted with permission. © The Water Research Foundation.

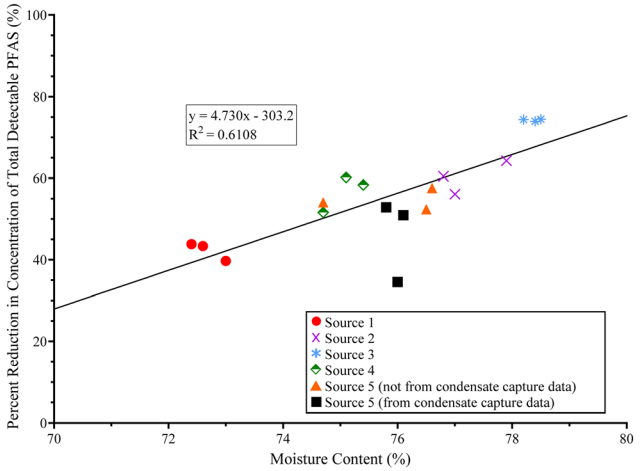


Fig. 2 Initial moisture content of biosolids is positively correlated to concentration reduction of total detectable PFAS (n = 18). Reporting limits were substituted in for non-detects. Reprinted with permission. © The Water Research Foundation.

of pure chemical PFAS at 100 °C indicates that volatilization alone is not a substantial removal mechanism for PFAS at this temperature. The percent reductions of PFAS in these biosolids drying experiments indicate that a removal mechanism other than (or in addition to) volatilization occurred.[36]

The removal of both volatile and nonvolatile PFAS *via* attachment to aerosols has been demonstrated for aeration tanks.[32,33] By extension, the formation of aerosols during boiling may occur *via* the bubble microtome effect.[37] Due to the tendency of PFAS to partition to the air–water interface (AWI), both small air bubbles underwater (*e.g.*, boiling bubbles) and small water droplets in the air (*e.g.*, aerosols) may contain significant amounts of PFAS, as both have high surface area to volume ratios; the surface area of an aerosol is effectively composed of the AWI.[37,38] Therefore, drying biosolids may incite the boiling of the water within and around the biosolids, thus leading to the generation of PFAS-laden aerosols.[26] An increase in the moisture content of biosolids may result in greater formation of aerosols, thereby giving rise to the observed positive correlation between moisture content and percent reduction of total detectable PFAS shown in Fig. 2. This trend supports data from previous research, which found a similar positive correlation between these variables.[26]

3.2 Incomplete mass balance on PFAS removal to the lab-scale dryer condensate

A fraction of the PFAS entered the lab-scale dryer condensate, yet the PFAS mass balance was not complete. On average, 16% of total PFAS from the wet biosolids were detected in the condensate. Additionally, the biosolids' detectable PFAS mass was reduced by an average of 46%. The "unaccounted" fraction of PFAS (represented by the white bars in Fig. 3 and 4a–c) corresponds to the difference between the initial PFAS

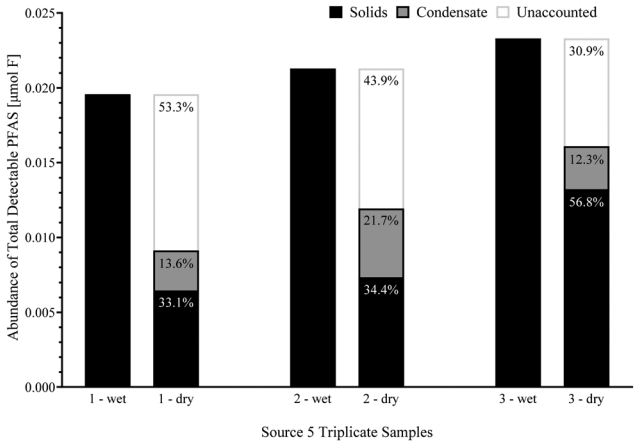


Fig. 3 Phase distribution of total detectable PFAS after drying (from source 5 triplicate samples). Abundance of PFAS was determined by normalizing reported concentrations to µmol F kg^{-1} solids or nmol F L^{-1} liquid, then multiplying that value by either the mass of solids or the volume of liquid in the impinger (Table S2). Only detected species were used for this figure (*i.e.*, reporting limits were not substituted in for non-detects). Reprinted with permission. © The Water Research Foundation.

mass in the wet biosolids and the combined mass detected in the dry solids and impinger methanol; the fate of this mass of unaccounted PFAS is unknown. On average, 43% of total PFAS as F remained unaccounted for across all triplicates, with a range from 31% to 53%.

Several reasons could contribute to explaining the unaccounted fraction. It is possible that PFAS were lost *via* sorption to part of the experimental apparatus since the entire surface area was not extracted for PFAS, *e.g.*, the tubing of the impinger system was not rinsed with methanol. If PFAS sorbed to the tubing, they did so by way of entering the gas-phase, which suggests that they should have been detected in the captured condensate but did not make it to the impinger. Additionally, a portion of unaccounted PFAS may stem from detectable PFAS transforming to undetectable PFAS, such as trifluoroacetic acid.[39] Approximately 10% weight loss of PFOA has been reported at temperatures of 100 °C, accompanied by the generation of volatile, fluorinated compounds.[36] OTM-50 was not employed in this research, and it is possible that non-polar volatile PFAS were generated as products of incomplete destruction but were not detected in the condensate.[40] Experimental data would be needed to validate this removal pathway. It is also possible there is an artifact arising from the heterogenous distribution of PFAS in biosolids samples, or that chemical extraction of PFAS from the dried solids samples was less efficient than extraction from wet solids samples.[41] Within the third triplicate specifically, the volume of methanol in the impinger was approximately eight mL less than the other triplicates by the end of the experiment. Although the reason for this difference in methanol volumes was not identified, it is possibly due to sample loss, thus reducing the mass of PFAS recovered in the condensate of the third triplicate in

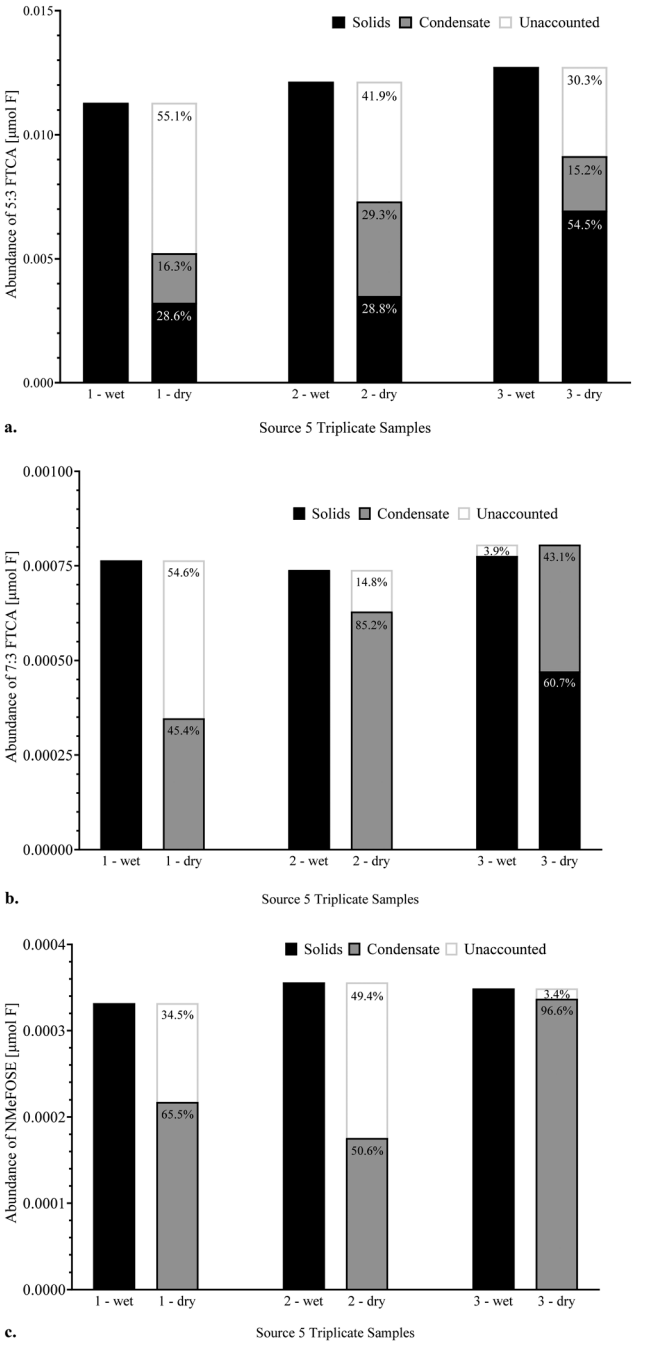


Fig. 4 a. Phase distribution of 5 : 3 FTCA after drying (from source 5 triplicate samples). b. Phase distribution of 7 : 3 FTCA after drying (from source 5 triplicate samples). c. Phase distribution of NMeFOSE after drying (from source 5 triplicate samples). Only detected species were used for this figure (*i.e.*, reporting limits were not substituted in for non-detects). Reprinted with permission. © The Water Research Foundation.

Fig. 3 and 4a–c. Despite the unclosed F mass balance, these results demonstrate a proof-of-concept that PFAS can be captured in dryer condensate.

Precursor species, including fluorotelomer carboxylic acids (FTCAs), were removed from biosolids during drying and captured in the condensate. Three species were

detected across all three triplicates of the captured condensate: 5:3 FTCA (Fig. 4a), 7:3 FTCA (Fig. 4b), and NMeFOSE (Fig. 4c); one species was detected only in one triplicate: NEtFOSE. More than three-quarters of the total PFAS in the condensate was attributable to 5:3 FTCA, across all triplicates. Though NMeFOSE and 7:3 FTCA comprised a smaller fraction of total PFAS across all phases than 5:3 FTCA, they exhibited substantial removal to the condensate.

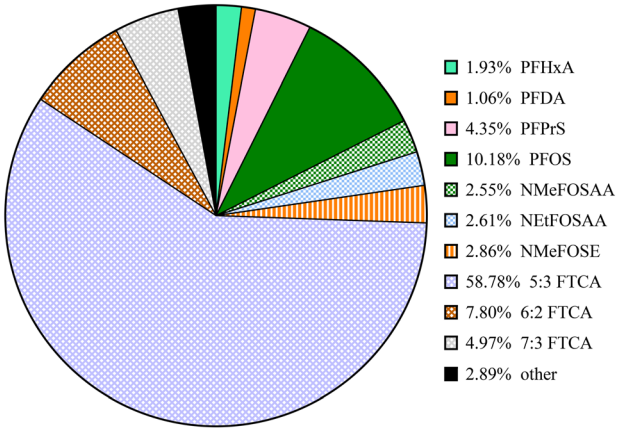
Not only are PFAS leaving with the gas-phase and appearing in the condensate, but in the case of 7:3 FTCA and NMeFOSE, some PFAS may not be detected at all in the dried solids. None of the dried biosolids triplicates had detectable levels of NMeFOSE, and only one had 7:3 FTCA, despite all three condensate triplicates revealing detectable amounts of both, as shown in Fig. 4b and c. In the sample with the lowest condensate fraction of 7:3 FTCA and NMeFOSE, nearly half of the amount present in the pre-dried biosolids was captured in the condensate. Since the temperature of the oven was approximately 105 °C, it is possible that this removal mechanism was physical (*i.e.*, removal *via* attachment to aerosols) rather than a direct phase transition (*i.e.*, volatilization); the relatively low volatility and high water solubility of FTCAs is consistent with the tendency to partition to aerosols.[33]

3.3 Species specific concentration reduction rates

On average, the concentration of total detectable PFAS in the pre-drying biosolids decreased by 57.6% after drying. The reduction in concentration of precursor species, on average, accounted for over 80% of this observed concentration reduction of detectable PFAS. Specifically, 5:3 FTCA contributed 60% to the reduction in concentration of total PFAS, and the top three precursors (5:3 FTCA, 6:2 FTCA, and 7:3 FTCA) contributed more than 71% (Fig. 5). Of the terminal species, the average reduction in concentration of PFOS was the greatest, at more than 10% of the total PFAS-as-F (Fig. 5). All of the ten species that contributed to more than 1% of the concentration reduction of total detectable PFAS are shown in Fig. 5 below. These data demonstrate that the majority of detectable PFAS concentration reduction observed during drying is attributable to a limited number of species. It should be noted that the capacity for a species to contribute to the reduction in concentration of total detectable PFAS is heavily dependent on the initial concentration of the species. For example, 5:3 FTCA constituted the largest proportion of the PFAS profile for all biosolids samples across all sites. Therefore, reducing its concentration will contribute significantly to the reduction in concentration of total detectable PFAS.

3.4 Role of FTCAs and quantification methods

The analytical technique used for this experiment detected 60 distinct species of PFAS, including all 40 species



Total = 57.6% (Average Percent Concentration Reduction of Total Detectable PFAS)

Fig. 5 Species that contributed at least 1% to the reduction in concentration of total detectable PFAS from biosolids. Reporting limits were substituted in for non-detects. Each species' contribution to the percent concentration reduction of total detectable PFAS from the biosolids was averaged for each site (n = 3), then the averages of each species across all sites were averaged (n = 5). Reprinted with permission. © The Water Research Foundation.

detected by EPA method 1633.[42] Five species that would have been excluded by method 1633 were detected in the experimental samples, making up, on average, 10.5% of the pre-dried biosolids' PFAS profiles and 1.6% of the post-dried biosolids' PFAS profiles. However, this experiment did not include detection of polyfluoroalkyl phosphoric acid diesters (diPAPs), which have been substantial PFAS species in other biosolids studies (54% and 56% of the total PFAS mass in biosolids, on average).[30,43]

Differing analytical techniques will detect different species of PFAS, which may influence concentration reduction calculations.[43] Some techniques exclude FTCAs in their analysis.[29] 5:3 FTCA was the most prevalent species of PFAS across all triplicates for all samples, including pre-dried solids, dried solids, and condensate. Had FTCAs not been detected in this set of experiments, the calculated average percent removal of total PFAS would have been 48% across all sites (instead of 58%). Furthermore, the exclusion of FTCAs from analysis dramatically reduces the detectable concentration of total PFAS to approximately 33% of the original values, as shown in comparing Fig. 6a and b. The detection (or lack of detection) of specific PFAS has a large influence on the results and conclusions drawn from an experiment.

Despite being the most prevalent PFAS species detected in biosolids from all sources, 5:3 FTCA is not a contaminant of direct industrial output; it is a metabolite from the transformation of other precursors, including 6:2 diPAP, 6:2 FTOH, and 6:2 FTSA, which may have extensive industrial applications.[44–50] 5:3 FTCA has been identified as the most highly concentrated PFAS present in landfill leachate.[46] Leachate being sent to the head of a WRRF may, therefore,

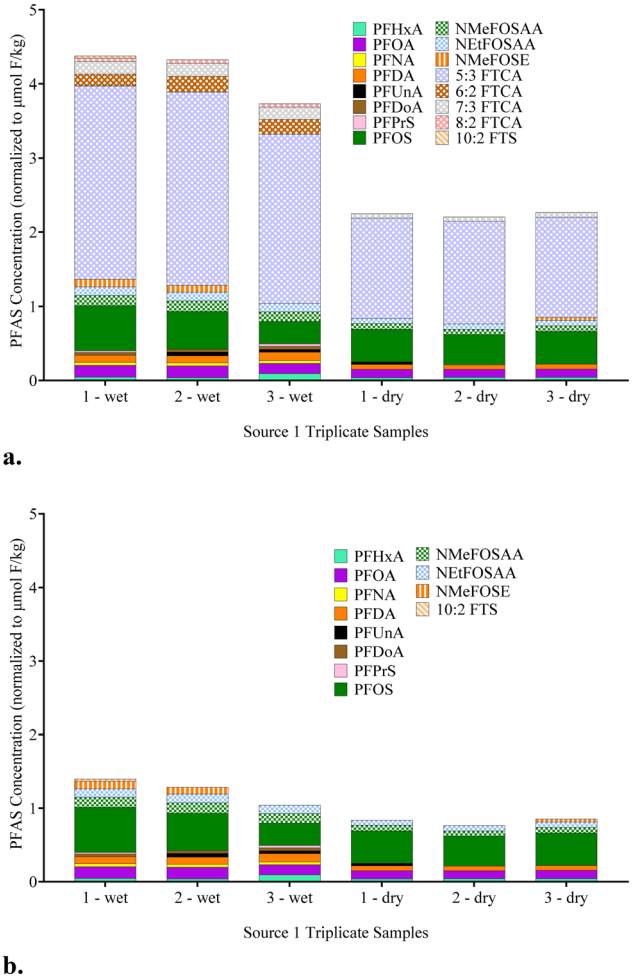


Fig. 6 a. Original PFAS profile of triplicates from the first site. b. PFAS profile of triplicate data from the first site excluding FTCA compounds (approximately 1/3 of the total PFAS from each sample is shown). Only detected species were used for this figure (*i.e.*, reporting limits were not substituted in for non-detects). Reprinted with permission. © The Water Research Foundation.

be a substantial source of 5 : 3 FTCA in wastewater treatment streams.

The FTCAs in land-applied biosolids may undergo transformation in food crops (*e.g.*, pumpkin and wheat) to terminal PFAS such as PFHxA, PFPrA, and PFOA.[51,52] Though FTCAs may not be regulated stringently, their presence is indicative of potential future contamination as they degrade and transform into perfluoroalkyl acid (PFAA) species.[51–55] This insight is especially crucial when paired with the condensate PFAS profile data that indicates 5 : 3 FTCA and 7 : 3 FTCA both left with the gas-phase during drying (Fig. 4a and b). The escape of these compounds, and their transformation potential, indicate that drying requires effective air pollution controls to reduce potential emission of not only these precursors, but their transformation products as well. Further research on the efficacy of current air pollution control technologies and possibly condensate treatment on PFAS-containing dryer emissions would be useful.[31]

3.5 PFAS treatment in condensate with PFAS from dryers

While drying could be a viable technology to reduce detectable PFAS levels in biosolids, treatment requirements for the resulting condensate from the drying process need to be further investigated. Condensate volumes from sludge dryers can vary significantly based on factors such as sludge type, dryer type, and operating conditions. Nonetheless, sludge dryers can produce substantial condensate volumes that may require further treatment and can be recycled to biological treatment portions of the plant.[56] In addition to conventional nutrients (*e.g.*, phosphorus and nitrogen), the condensate can also contain PFAS.[29] The mobilization of PFAS from the biosolids into the liquid phase (*i.e.*, condensate) represents an opportunity to disrupt the PFAS contamination cycle at WRRFs.[26]

To break this cycle, a separation step followed by destruction could be an efficient method to drastically reduce PFAS concentrations.[13] One possible separation technique is foam fractionation, where air bubbles are utilized to separate surface-active molecules (*i.e.*, PFAS). Through this process, PFAS are concentrated in a foamate solution that can be directed for subsequent destruction. This step offers a dual advantage as the PFAS concentrations in the foam can be enriched by a factor ranging from 100 to 100 000, and the volume requiring treatment is significantly smaller compared to not using foam fractionation.[57]

To date, various destruction techniques have been utilized to eliminate PFAS, each with its own distinct advantages and disadvantages, indicating a need for a customized approach for each WRRF.[58] Among other technologies, field installations of supercritical water oxidation (SCWO) and electrochemical oxidation (EO) have recently garnered significant attention. In SCWO, water is heated above its critical point to create a supercritical state where it exhibits properties of both a liquid and a gas. This high-temperature and high-pressure environment allows for the complete oxidation of organic contaminants, like PFAS, breaking them down into byproducts such as carbon dioxide, water, and mineral acids.[59] During EO, an electrical current is applied to the contaminated water or solution, causing oxidation reactions to occur at the electrode surfaces. These oxidation reactions break down the PFAS compounds into less harmful byproducts through electrochemical processes.[60] Combining foam fractionation with either SCWO or EO could offer a promising solution to prevent the re-mobilization of PFAS back into the biological treatment section of a WRRF after removal of the PFAS from the biosolids stream. The net life cycle cost of this flow diagram (drying with PFAS treatment of the condensate) may be advantageous when compared to PFAS destruction in the full biosolids stream.

3.6 Future perspectives

The current study has revealed that PFAS transport to dryer condensate during biosolids drying is feasible. Due to the heterogeneity of biosolids samples, it is necessary to analyze

the PFAS profiles of dryer condensate from different biosolids samples to establish the reproducibility of these experiments. Additionally, a more robust experimental design is required to further elucidate PFAS fate, including their potential for transformation and removal during biosolids drying. Effective detection of precursor compounds, especially diPAPs, is key to developing a more comprehensive understanding of PFAS fate.[61] Future work should include the quantification of diPAPs in sample analysis and the extraction of PFAS from laboratory equipment with a methanol rinse. Augmenting targeted detection methods with analytical tools such as the total oxidizable precursor (TOP) Assay may also improve the understanding of PFAS fate.

4.0 Conclusions

This research confirms that concentrations of detectable PFAS in biosolids are reduced during drying. Furthermore, the majority of concentration reduction is attributable to a reduction in precursor species, particularly 5:3 FTCA. Therefore, reduction in concentration of PFAS in biosolids during drying may provide an avenue by which PFAS can be captured and destroyed, thus mitigating contamination cycles. However, should biosolids drying not be paired with some form of air pollution control and/or condensate treatment, dryer air emissions or condensate may contain precursors such as 5:3 FTCA, 7:3 FTCA, and NMeFOSE that can then transform into PFAAs.[62] While the vapor capture technique utilized herein revealed removal to condensate of some PFAS, more research is needed to more fully close the F mass balance from drying of biosolids, by either confirming transformation products or detecting previously undetectable PFAS. Measurements of PFAS concentration reduction are constrained by the chosen detection method.

This research aimed to help fill the gap in the literature on the effect of drying on biosolids' PFAS profiles, including concentration reduction percentages and phase distribution, and to correlate PFAS concentration reduction with initial biosolids moisture content. Moisture content of the initial biosolids sample correlated to greater reduction of total detectable PFAS concentration, indicating that aerosolization during biosolids drying is a potential avenue by which PFAS are being removed.[26,37] This process circumvents the requirement for high temperatures associated with volatilization and mineralization, and may be a substantial removal mechanism for PFAS in biosolids dryers. By developing a robust understanding of how PFAS concentrations are reduced during biosolids drying and where exactly they end up, full-scale facilities can be optimized to control and increase the removal of PFAS from biosolids during drying and to capture and treat the removed PFAS. Overall, drying could be used to increase the accessibility of biosolids land application.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI).

Supplementary information is available and describes the reactor setup, PFAS analytes quantified, and PFAS profiles of wet and dry biosolids samples. See DOI: https://doi.org/10.1039/d5ew01120e.

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